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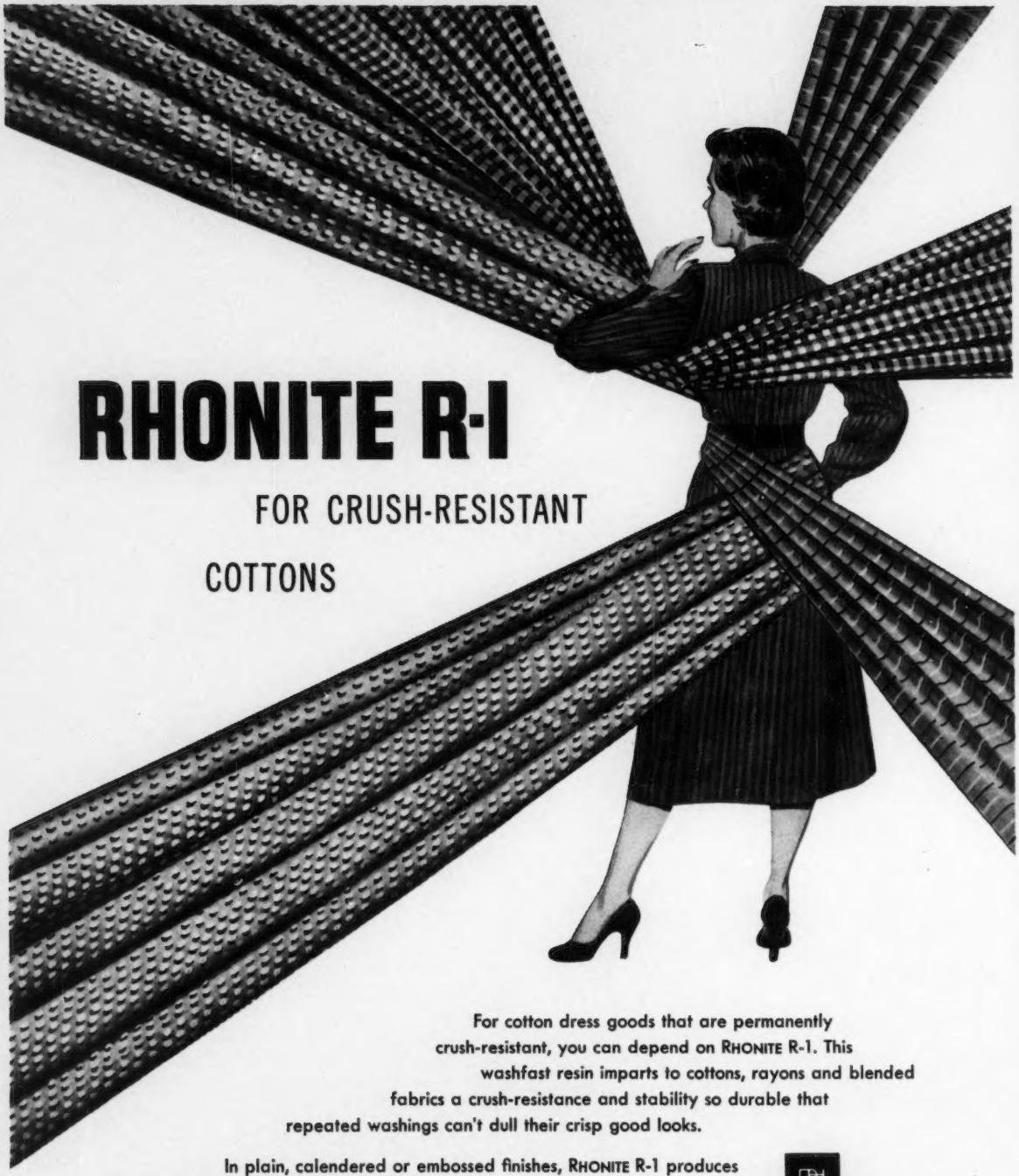

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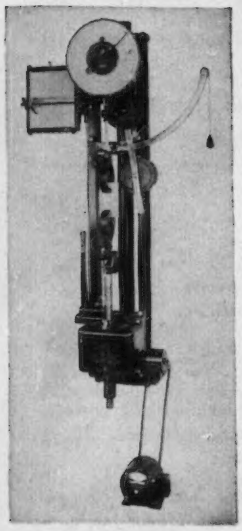

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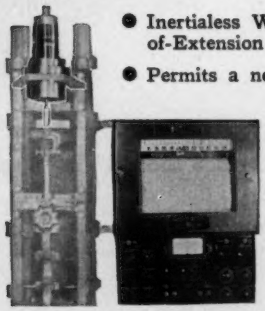

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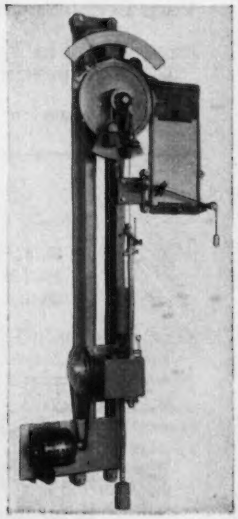

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TEXTILE RESEARCH JOURNAL

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Some Fundamental Questions Concerning the Static Electrification of Textile Yarns: Part II *

V. E. Gonsalves and B. J. van Dongeren

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Introduction

Under certain circumstances during the manufacture and processing of yarns, especially synthetic yarns, electrostatic charges due to friction occur on the yarn, and these may exert an unfavorable influence on the manufacturing process.

For example, during the manufacture of spun rayon, the rolls, slivers, and intermediate yarns become more voluminous due to the mutual repulsion of the charged fibers, in consequence of which the production units become lighter. The greatest difficulty occurs on the carding machine. The mutual repulsion of the fibers results in an incohesive fleece; attraction of the fleece by the metal parts of the machine gives a bulging, undulating fleece, with greater chance of breakage. Sometimes the attraction of the fibers by the doffer comb is so great that no fleece can be formed.

On the drawing frames static charges cause coiler tube chokings and roller lappings.

During the warping of nylon, Enkalon, and similar yarns on the warping machine, the threads diverge when the machine is stopped, as this decreases the yarn tension; this results in a larger width of the sheet of threads. This causes great difficulties on beaming-off.

During weaving, the weft thread is carried along a little by the returning reed, in consequence of which this thread does not remain stretched between the warp threads; this causes an irregular fabric. On beam-sizing nylon, the threads of the sized beam diverge after drying, which results in slacker threads, which may break subsequently, and an irregular fabric.

Various means for preventing or counteracting all these difficulties are being developed. By applying so-called "antistatic" finishing agents, "antistatic" thread guides, and "antistatic" roller linings, the occurrence of the charge can be prevented. It is also possible to eliminate any charge formed with the aid of so-called "static eliminators" before difficulties can be caused.

In the literature various measuring methods are mentioned, both for measurements on laboratory arrangements and on processing machines.

Lehmicke [3] described a method in which textile material is rubbed between rubber gloves and is then thrown into a metal beaker which is connected to an electrometer.

Ott [5] described a method in which an insulated plate is rubbed over textile material. After some time the contact between the plate and the textile material is interrupted, and the potential of the plate is measured with a valve voltmeter.

Hayek and Chromey [1] described a method in which the textile material is fastened on an aluminium cylinder. The textile material is rubbed on a brass strip which is also covered with textile material and presses against the cylinder. The charge induced on the aluminium cylinder is measured.

Medley [4] drew yarn over a platinum wire with the aid of an insulated rod. The potential of the wire is measured with a Lindeman electrometer.

* Part I appeared in the October, 1953, issue of TEXTILE RESEARCH JOURNAL.

Presented at a meeting of the Fiber Society in New Orleans, La., Apr. 22, 1953.

Pieper [6] measured the charge of a metal thread guide with an electrometer; the thread passing over this thread guide is rubbed beforehand with glass rods and the like.

All these measurements can be termed laboratory measurements.

Stoll [7] took measurements on card fleeces. For this purpose a conductor was placed near the fleece, and the reverse charge induced on this conductor by the charged fleece was measured with an electrometer.

Keggin, Morris, and Yuill [2] described another measuring method applied in practice. The sliver which is formed from the fleece and leaves the pressure rollers is collected in a Faraday cage. The charge of the cage is measured with a valve voltmeter.

The following general remarks can be made about the above-mentioned measuring methods. As regards the direct measurements, in principle a distinction can be made between (*1*) measurements of the potential and (*2*) measurements of the charge.

The measurement of the potential can be carried out directly or capacitatively; however, neither method gives a correct value for the charge of the examined material, the capacity of an insulator being an undefined concept. The measurement of the charge can be divided into: (*1*) measurement of the total charge; (*2*) measurement of part of the charge.

If a scanning device is used, only part of the charge is measured. Moreover, there is a great chance that during scanning a charge is generated, for here, too, friction actually occurs.

Of course the measurement of the total charge gives the best result; from this it follows how many units charge are present per unit length or weight of yarn, sliver, and the like.

In our opinion the measurement of the charge is preferable to measurement of the potential for the following reason. The difficulties caused by the static electrification find expression as forces, and these forces are governed by Coulomb's law—*i.e.*, two charged spherical conductors at a distance a exert a force on each other which is only dependent upon the charge of the conductors and not on the potential (assuming that the radii of the conductors are much smaller than the distance a).

A fortunate circumstance is that a measurement of the charge is more accurate and can be made in a simpler way than a measurement of the potential.

In order to be able to measure the charge of the yarn, an electrometer is required.

We shall first describe the valve voltmeter with electrometertriode which we have developed for this type of measurement. Then follows a description of the entire measuring arrangement, by means of which the static electrification of yarns can be measured.

Measuring Apparatus

Valve Voltmeter with Electrometertriode

A schematic drawing of this electrometer is shown in Figure 1. This meter makes it possible to measure both positive and negative charges. The component parts are described below.

EMT is an electrometertriode with microammeter *M* in the plate circuit. The plate voltage can be adjusted with the aid of the potentiometer R_1, connected to the 4.5-volt battery via the switch S_2. The filament is fed by a 4-volt accumulator via the switch S_1.

S_6 is brought in position *O* and S_5 in position *D*; after press-button S_4 has been pressed down and S_3 has been closed, the grid will obtain the voltage,

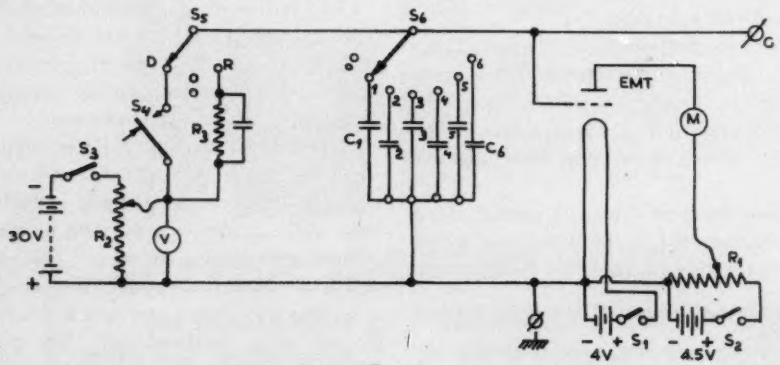

FIG. 1

which can be read on voltmeter V. This will influence the deflection of the pointer of M; with the aid of the potentiometer R_2 the calibration characteristic of the valve voltmeter can now be determined.

In order to be able to take a measurement, first a (suitable) condenser (*e.g.*, S_6 in position *1*) is chosen and a certain negative potential (*e.g.*, -1 volt) is given to the grid by pressing press-button S_4 for a moment while S_5 is in position D and the potentiometer R_2 has been adjusted at 1 volt.

If now a certain charge, Q, is imparted to G, the potential difference between the grid and the earth will change by an amount $\Delta V = Q/C$, where C is the total input capacity of the measuring apparatus. The potential difference ΔV follows from the reading of the microammeter and the calibration characteristic. Since C is also known, the magnitude of the charge Q follows from C and ΔV.

Positive charges cannot be measured in the same way, because the grid of the triode would then become positive and thus would attract electrons. For that reason, care is taken that, before the measurement, the grid and the adjusted condenser have such a negative charge that after imparting the positive charge to G the grid still remains negative (at least -1 volt).

Of course the charge brought on G should not leak away; it is for this reason that we use an electrometertriode, in which the grid current in the negative grid voltage region is negligibly small.

The Measuring Arrangement

The arrangement for measurements on yarns, indicated schematically in Figure 2, consists of the parts described below.

W_1 is a well insulated, stainless-steel friction-causing member in the form of a screw thread. The angle of wrap of the yarn on this friction-causing member is adjustable (not indicated in the figure) and can be read on a graduated arc.

The friction-causing member is connected with terminal G of the valve voltmeter, described in the preceding section and schematically indicated in Figure 2 by LM.

M_1 is a yarn-pulling-off mechanism and M_2 is a take-up spool with slip clutch. The speed of the yarn can be adjusted by varying the number of revolutions of M_1. With the aid of W_2 the yarn can be given a certain pre-tension. This tension can be read from the tensiometer TM_1. TM_2 indicates the thread tension after the friction-causing member W_1. *St.El.* is a static eliminator, which takes care that the thread arrives without charge on the friction-causing member W_1. This is necessary for the quantitative determination of the charge which is generated by the friction-causing member W_1. The distance between *St.El.* and W_1 is approximately 1 m.; in this way care is taken that *St.El.* does not remove the charge generated by W_1. Let us suppose that the yarn is charged negatively during its passage over W_1; then W_1 itself becomes positive. Now the time required for reducing the preadjusted negative voltage of condenser C and grid (see the preceding section) by a certain amount V is measured. From this time, t (sec.), the speed of the yarn, v (cm./sec.), the capacity, C ($\mu\mu F$), and the reduction, V, in voltage of C (volts), it can be calculated, according to the equation given below, how many units charge ($\mu\mu$Coulomb) are present per cm. of yarn at the given pretension of the yarn, the relative humidity of the air, and the given wrap of the element W_1:

$$\text{charge} = \frac{CV}{vt}\ \mu\mu C/\text{cm. of yarn length.}$$

The sign of the measured charge is opposite to that of the charge on the yarn.

If the charge on the yarn is very high, and therefore the measured time very small, even when the largest condenser is used, so that the measurements are not sufficiently accurate, then another method is applied.

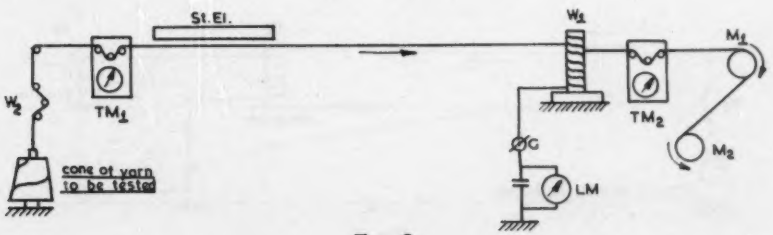

FIG. 2

For this purpose, S_3 (see Figure 1) is adjusted in position R and S_6 in position O, so that the grid of the electrometertriode is connected to the sliding contact of the potentiometer via a high resistance R. The charge to be measured will now leak away via this high resistance, and will thus cause a voltage drop across the latter. From the reading of the meter M and the calibration characteristic this voltage drop can be ascertained, and from this voltage drop (and the resistance R and the speed of the yarn) the charge per cm. of yarn can be calculated.

Check on the Measuring Method

In order to show that the absolute value of the measured charge on W_1 is as large as that of the charge on the yarn, the arrangement shown in Figure 3 was set up.

Sp is a spool on an insulated spindle which rotates in a Faraday cage, K, also mounted on an insulating stand. The yarn, charged by the friction over W_1, is wound on the spool *Sp*, and induces on the outer surface of the cage K a charge of the same numerical value and of the same sign as the charge on the yarn.

W_1 and K were both connected to the terminal G of the valve voltmeter, *LM*.

When the static eliminator had been provided, no electrostatic charge was measured by *LM*, so the charges of W_1 and K neutralized each other, proving that the measuring method was correct. Small fluctuations of the meter needle around the equilibrium position indicated inhomogeneities in the thread.

When the *St.El.* was removed, the meter indicated the charge which the yarn had obtained due to the friction in the zone before W_1 (*e.g.*, at W_2). Thus, this method also served to check the operation of the *St.El.*

When friction-causing members made of nonconductive material are used, it is not possible to effect a connection to the meter. In such cases the charge is measured with the Faraday cage. Then the sign of the measured charge is the same as that of the charge of the yarn.

Points to be Noted during the Measurements

Finishing agents and their distribution on the thread.—When a finished thread passes through the measuring apparatus, the measurements are not reproducible until all parts of the measuring arrangement along which the thread runs are covered with that finishing agent. In order to keep the time required to effect this as short as possible, the thread is moved along the same path as much as possible. The thread should always pass through the same groove of the stainless-steel screw thread. After an irregularity in the thread has passed (a knot or fluff), the same rule applies—wait until all parts are again sufficiently covered with finishing agent.

Irregular distribution of the finish on the yarn also prevents reproducible observations. This irregular distribution is usually a consequence of the way in which the examined yarn has been wound or coned. For example, when coning a skein, the coning machine removes finishing agent from the first part. On measuring the static charge, the measuring arrangement removes finish from the last part of the skein. Therefore, only the part in between has satisfactory, regular distribution of finishing agent. Only the measurements on this part appear to be sufficiently reproducible.

Therefore, between two measurements on yarns with different finishing agents, all parts along which the thread passes must be cleaned thoroughly with a 67.5% benzene-32.5% alcohol mixture.

Traces of other finishing agents on the coning machine or the measuring arrangement also prevent reproducible observations.

The measuring process.—When measuring small charges, a condenser of small capacity must be inserted. Then the meter is sensitive to the so-called "hand capacity effect." Walking and other super-

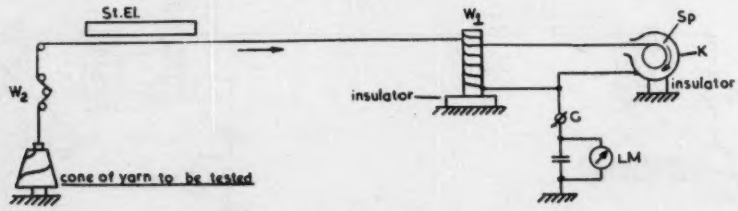

FIG. 3

fluous movements of the observer near the measuring arrangement should therefore be avoided.

When using a static eliminator which is operated by high voltage, the measuring instrument may be disturbed; also for this reason the eliminator must be set up as far as possible from the friction-causing member and the charge meter.

Orienting Experiments

A few preliminary measurements will now be described, which will show what may be expected as regards the static electrification of yarns. Moreover, interpretation of the experimental results with the aid of the theoretical considerations described in Part I of this series will be attempted.

In all the measurements described below the thread was rubbed against the stainless-steel friction-causing member. All threads for a given measurement were conditioned at the same relative humidity.

Measurement I

A finished rayon thread 300/60 was examined. The running speed was 50 m./min., and the relative humidity was 17%.

The charge of the thread was measured at three angles of wrap: 10°, 50°, and 90°.

The measurement was taken at various thread tensions before the friction-causing member; since the thread tension after the friction-causing member was also measured, the increase in thread tension, Δp, caused by the friction member was also known.

Figure 4 shows the results of the measurement of thread tension; Δp is plotted as a function of the pre-tension, p_v.

We shall consider the case $\Delta p = 2$ g. From Figure 4 it is seen that at an angle of wrap of 10° this Δp occurs at a pre-tension of 19 g., at 50° at a pre-tension of 4 g., and at 90° at a pre-tension of 2 g.

Therefore, the average thread tension over the friction-causing member is approximately 20 g. at an angle of wrap of 10°, 5 g. at 50°, and 3 g. at 90°.

At the three angles of wrap of 10°, 50°, and 90°, the normal pressures exerted on the friction-causing member per unit thread length * are then in the proportion 20:5:3 (the lengths over which the thread contacts the friction-causing member are in the proportion 1:5:9).

The results of the measurement of the charge of the thread as a function of Δp are plotted in Figure 5. For $\Delta p = 2$ g. it is seen that the generated charge in the three examined cases is practically equal (6 to 8 $\mu\mu$C/cm.).

The result of this experiment is that with shorter lengths over which the thread contacts the friction-causing member, the normal pressure required to obtain the same amount of charge per cm. of thread length is proportionately greater.

* V. E. Gonsalves, TEXTILE RESEARCH JOURNAL 20, 29 (1950).

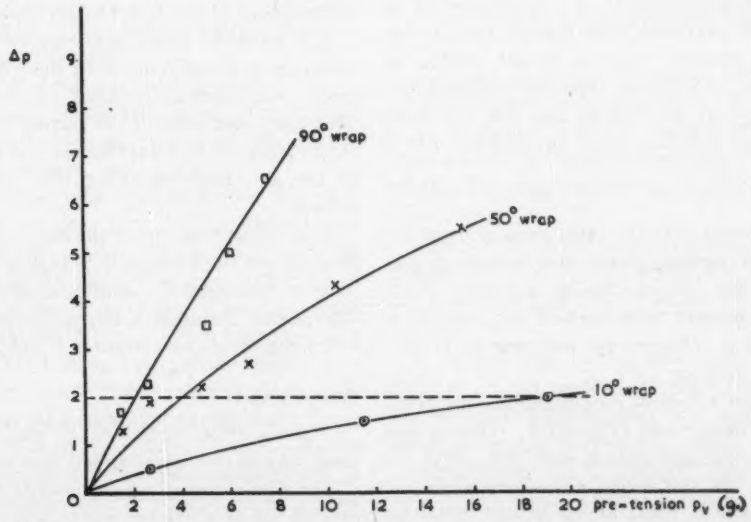

FIG. 4

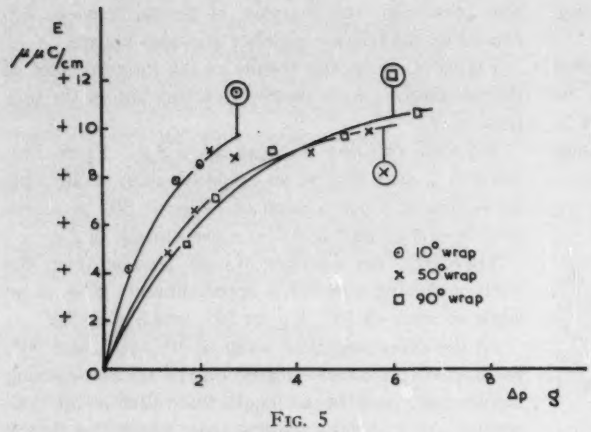

FIG. 5

In Part I of this series it was pointed out that in general the surface of an insulator will not make good contact with a metal surface; contact will be considerably improved by rubbing. Therefore, at low normal pressure the longer the distance over which the thread element dx has been in contact with the metal (the longer the time of rubbing), the better the contact. Thus, it can be understood that at low normal pressure and a large angle of wrap the same charge can be generated as at high normal pressure and a small angle of wrap.

From this measurement the following conclusion may be drawn: In order to keep the electrostatic charges on a thread as small as possible (at a certain tension of the arriving thread), it is advantageous to reduce the length over which the thread contacts the friction-causing member (*e.g.*, a thread guide) as much as possible. Of course, this only applies if the maximum charge of the thread has not yet been reached (see Part I of this series, pp. 712 and 717).

Measurement II

The rayon thread 300/60 (see measurement *I*) was examined at various speeds and various angles of wrap round the friction-causing member. Two series of measurements were carried out: one at a pre-tension of 5 g. (Figure 6) and one at a pre-tension of 10 g. (Figure 7).

During the first series of measurements the relative humidity of the air was 17%–23%. During the second series of measurements it was 14%–15%.

The results of the first series of measurements are presented in Figure 6, those of the second series in Figure 7.

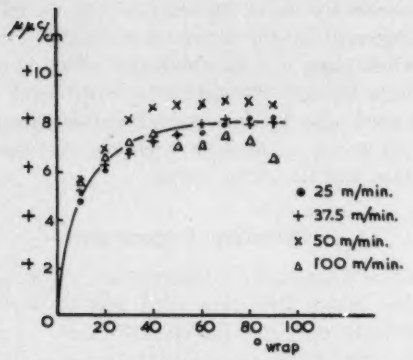

FIG. 6. *Rayon 300/60; pre-tension 5 g.*

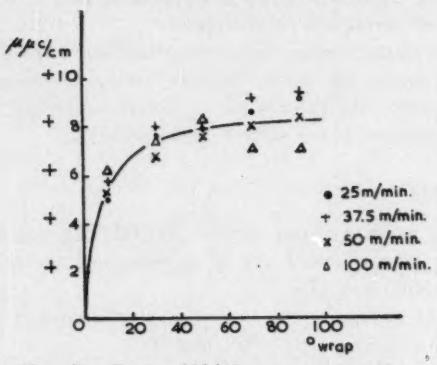

FIG. 7. *Rayon 300/60; pre-tension 10 g.*

The following observations may be made:

(*1*) The electrification of the thread is practically independent of the speed of the thread.

(*2*) In both cases the maximum electrification amounts to 8 $\mu\mu C$/cm., even though the relative humidity was about 20% during the first series of measurements, and about 15% during the second series. Apparently, in this experiment the relative humidity of the air (between about 10% and 25%) is not critical.

(*3*) In the zone where the maximum electrification has not yet been attained, a higher normal pressure (higher pre-tension) should, according to our considerations, result in a larger charge; however, this is not apparent from Figures 6 and 7.

Measurement III

A sized nylon thread 45/15 was examined in the same way as the rayon thread (see measurement *II*).

The first series of measurements (Figure 8) were carried out at a pre-tension of 4 g.; the relative humidity was 20%–25%. The second series (Figure

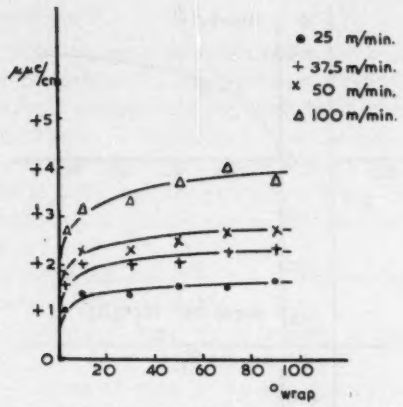

FIG. 8. *Nylon 45/15; pre-tension 4 g.*

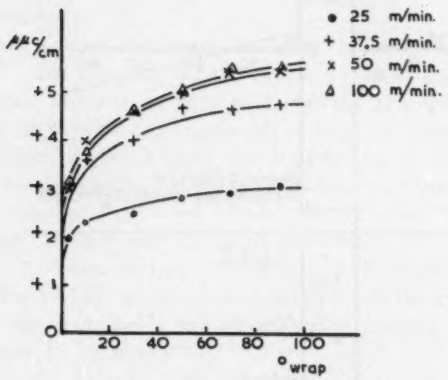

FIG. 9. *Nylon 45/15; pre-tension 10 g.*

9) were carried out at a pre-tension of 10 g.; the relative humidity was 19%–22%.

The following observations may be made:

(*1*) In this case the electrification is dependent upon the speed of the thread; in general, the electrification increases with increasing speed.

In our opinion the best explanation of this phenomenon is that the size of the nylon thread was somewhat conductive. After the electrification (the formation of the double layer), recombination of + and − charges can occur again due to leakage in the dielectric of the double layer, which is regarded as a "condenser." This leaking back of the charge is a relaxation phenomenon. If the relaxation time τ is of the same order of magnitude as the time t_a for which a thread element dx was in contact with the friction-causing member, then the electrification of the thread will depend upon this speed in the following way: increasing speed will result in increasing

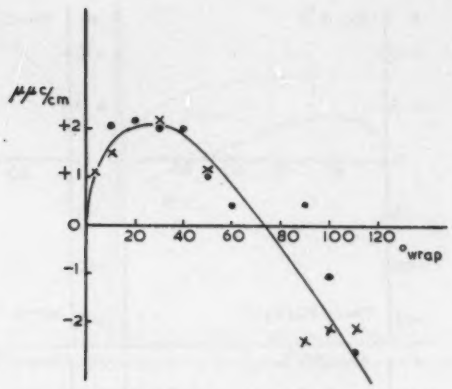

FIG. 10. *Rayon 300/75.*

charge, which is in agreement with the measuring results. In Figure 9 (lower relative humidity and higher pre-tension) it is seen that at the two highest speeds there is little difference in electrification; thus, it is possible that in these two cases the time of contact, t_a, of a thread element dx with the friction-causing member was considerably shorter than the relaxation time, τ.

(*2*) Increasing the normal pressure (higher pre-tension) results in higher charge.

Measurement IV

An unfinished rayon thread 300/75 was examined. The charge was measured as a function of the angle of wrap. The thread speed was 50 m./min.; the pre-tension was 5 g.; and the relative humidity of the air was 16%.

Although in general it appears to be impossible to take satisfactory measurements on unfinished threads, it so happened that the surface properties of this thread were so constant over long zones that on repeating the measurements practically the same results were found.

The results of the measurements are presented in Figure 10; plots for the first series of measurements are shown as dots, and those for the second series as crosses.

It is seen that in this case there is a reversal of sign; at a small angle of wrap (low normal pressure) the thread is positive, and at a large angle of wrap (high normal pressure) the thread is negative.

Apparently, an "activated" thread is concerned here. The theory of the phenomenon was discussed in Part I of this series.

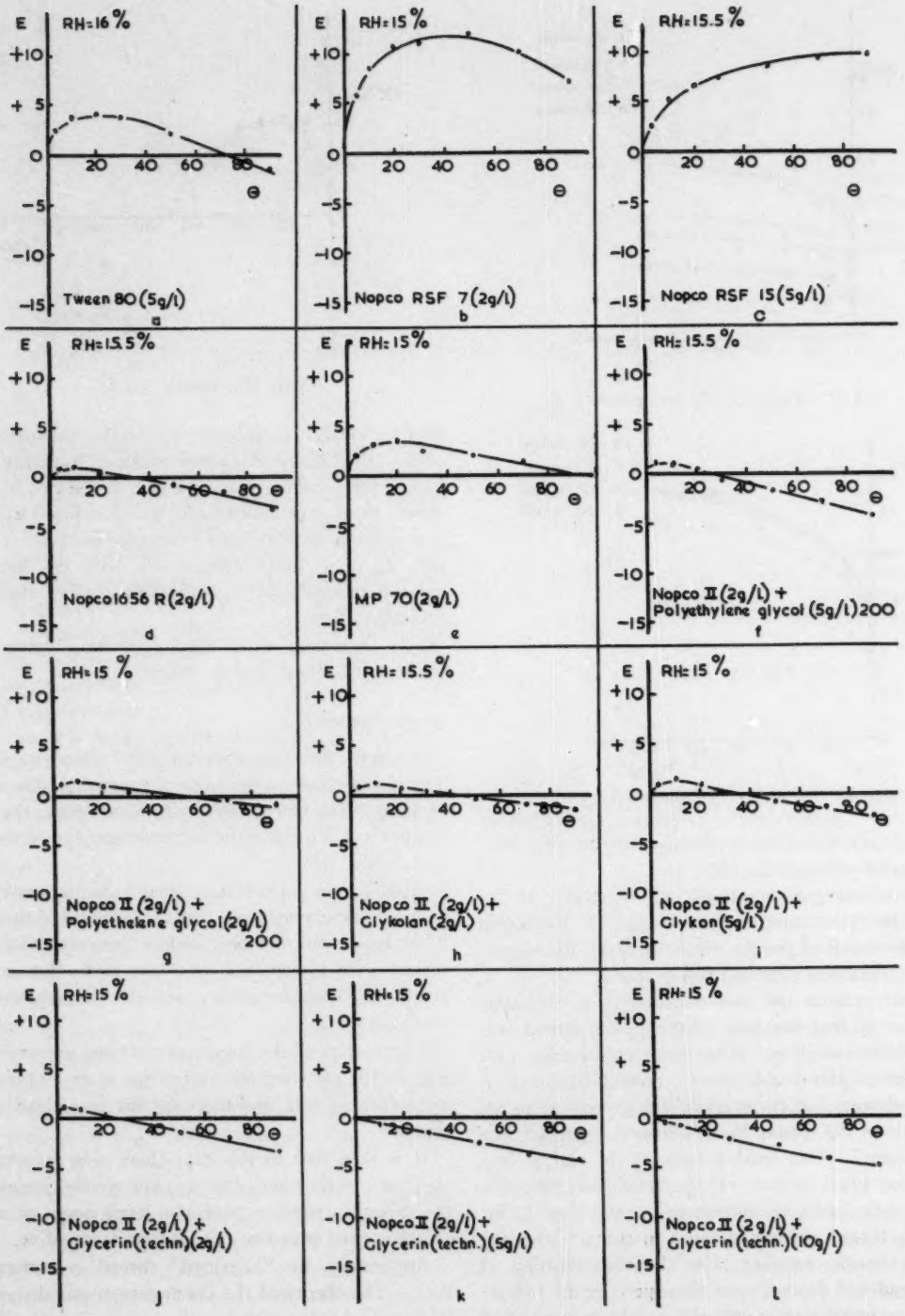

FIG. 11. *The static electrification, E ($\mu\mu C/cm.$), of a rayon thread 300/60 treated with various finishing agents, against stainless steel at different angles of wrap, θ. The thread speed was 50 m./min., and the pre-tension 5 g.*

Measurement V

In order to get a preliminary survey of the behavior of various finishing agents, a thread 300/60, treated with various finishing agents, was examined.*

The charge, E ($\mu\mu C$/cm.), was measured as a function of the angle of wrap, θ. The thread speed was 50 m./min., and the pre-tension 5 g. The relative humidity of the air was as indicated in Figure 11 (*a* to *l*).

The measured $E = f(\theta)$ can be represented schematically as indicated in Figure 12:

(*1*) Curves of type *a*; E reaches a maximum value.

(*2*) Curves of type *b*; at low normal pressure (small angle of wrap) the thread is positive; at high normal pressure (large angle of wrap) the thread is negative.

(*3*) Curves of type *c*; the thread is always negative. We observe that the case opposite to *2* (low normal pressure (θ is small)→thread is negative, and high normal pressure (θ is large)→thread is positive) does not occur (at least not up to now). These phenomena are strikingly similar to what was expected on theoretical grounds for so-called "activated" threads (see Part I of this series).

(*4*) Curves of type *d* (indicated by a dashed line in Figure 12); these are the cases in which the finishing agent obviously has an antistatic influence (see Figure 11 (*g*, *h*, *i*)).

Measurement VI

In this experiment the static electrification of the 300/60 rayon thread was not only ascertained at different angles of wrap, but also at different relative humidities of the atmosphere (the speed of the thread was again 50 m./min., and the pre-tension 5 g.).

Figure 13 shows the experimental results. A horizontal row of graphs relates to one particular finishing agent. In the graph on the extreme right the charge has been plotted against the angle of wrap; in the three graphs to the left of this one the charge has been plotted against the relative humidity for three different angles of wrap (10°, 50°, and 90°). From the last three graphs we can extrapolate to the relative humidity 0%; these values have been indicated by dashed curves in the graph on the extreme right.

It can be seen that in the case of the finishing agent MP 63 E 8a, at 40% relative humidity a sign change occurs; otherwise all positive states of charge at small angles of wrap show a tendency to become negative at large angles of wrap.

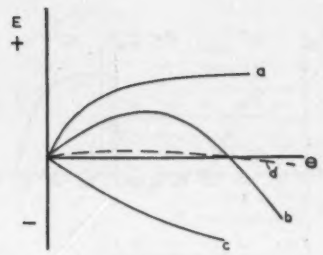

FIG. 12

From the three graphs on the left it follows, however, that at constant angle of wrap the charge can change from negative to positive at increasing humidity of the air; therefore, the surface properties of the finished cellulose thread apparently change at increasing moisture regain.

The last measurement (Na-oleate) gives the picture for a good antistatic finish; the charge remains low at all possible angles of wrap and relative humidities.

Measurement VII

The measurement described under *VI* was repeated, but with an Enkalon thread 50/10. The results are given in Figure 14.

Although these graphs resemble those of Figure 13, certain differences can be observed.

The principal difference is that in the case of Enkalon there is no sign change at increasing angles of wrap; at least, it has not been found yet.

Further, it can be observed that *if* at low relative humidity negative charges occur, these charges do become positive at increasing relative humidity, but not to the same degree as in the case of rayon.

The two last measurements relate to transformer oil KB 30, used as a finishing agent. Transformer oil is distinguished from normal textile oils by a very high resistivity and a very high dielectric strength. Our results show that, from the viewpoint of antistatic action, transformer oil is inferior; not only do considerable charges occur, but also the static charge, at 70% relative humidity and upwards, does not become negligibly small. (This in contrast with all other oils, which no longer have any charge at this percentage.)

The measurements relating to the finish Shell Anti-

* These samples were prepared by Dr. Hennemann in our textile department.

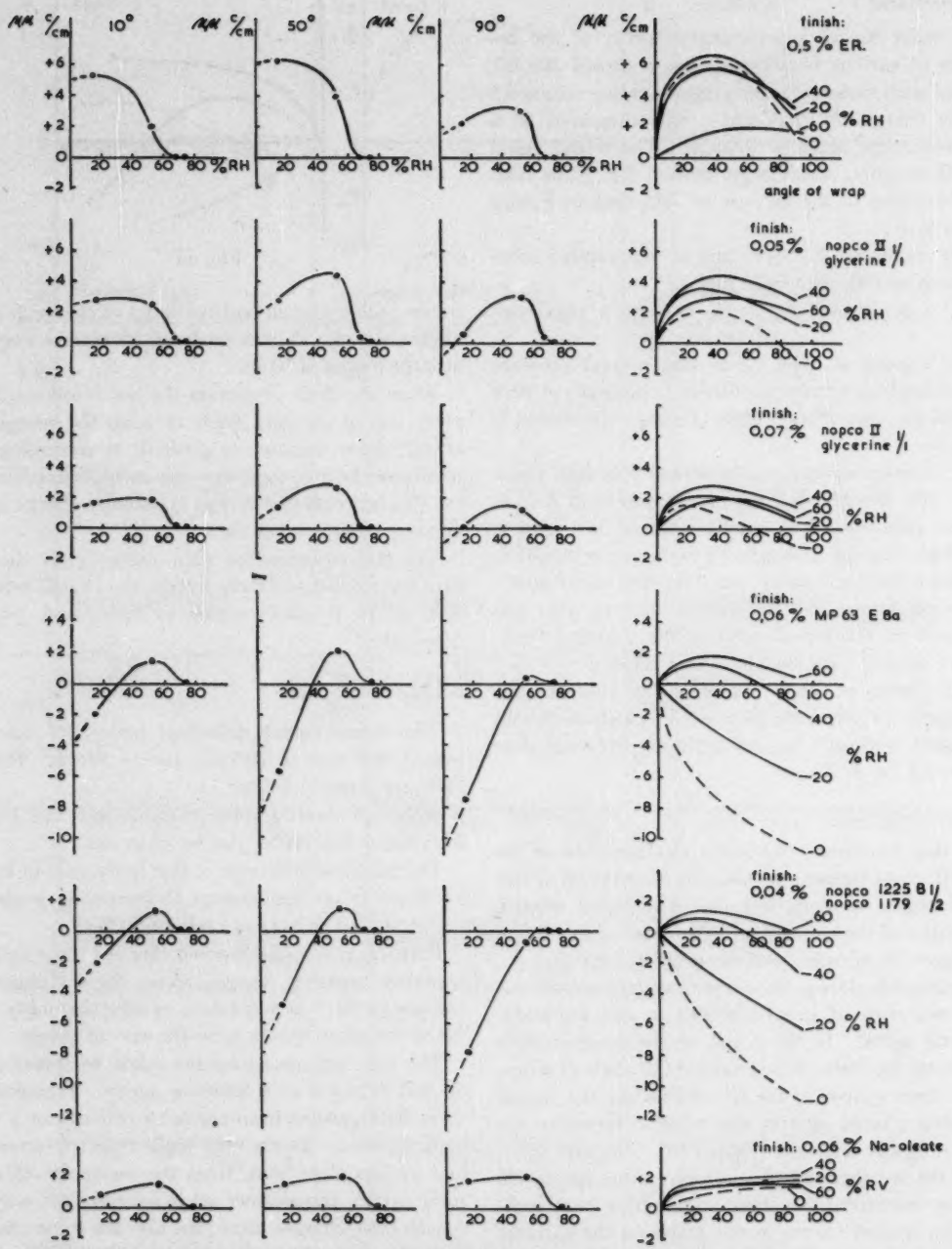

FIG. 13. *The static electrification (μμC/cm.) of a rayon thread 300/60 treated with various finishing agents, against stainless steel at different angles of wrap and different moisture contents. The thread speed was 50 m./min., and the pre-tension 5 g.*

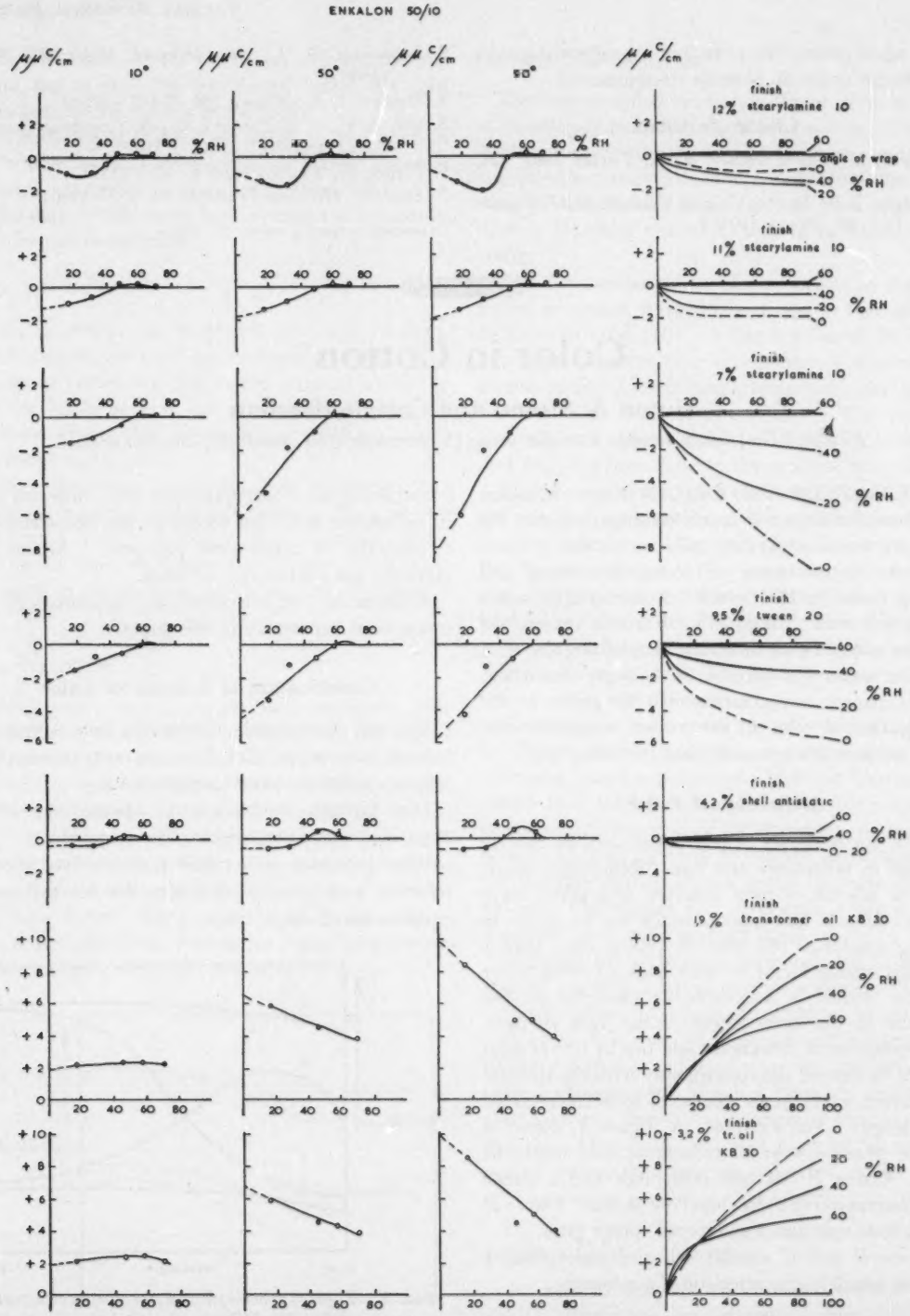

FIG. 14. *The static electrification (μμC/cm.) of an Enkalon thread 50/10 treated with various finishing agents, against stainless steel at different angles of wrap and different moisture contents. The thread speed was 50 m./min., and the pre-tension 5 g.*

static again present the picture of a good antistatic—a low charge under all possible circumstances.

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Color in Cotton*

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THE NATURE AND ORIGIN of color in cotton have been the object of much investigation, and the literature contains varying opinions on this subject. With the improvement of cotton harvesting and ginning methods, the "grade" of cotton approaches synonymity with "color" [7]. For this reason, the need for obtaining an understanding of the source of color in cotton has become increasingly important. In this paper, consideration will be given to the classification of color in raw cotton, especially that which persists after conventional bleaching [3].

Definition of Color

Color, for purposes of this paper, will be defined in terms of reflectance and hue. Reflectance measures the amount of light reflected at a given wave length. Hue is that quality which we recognize in terming a color as red, blue, or yellow, etc. Hue is the main quality factor in color, and it is that factor of color which is noticeable as changing in the spectrum as the wave length of the light changes. The evaluation of reflectance and hue in cotton samples can be carried out conveniently with the spectrophotometer, which gives a curve of reflectance *versus* wave length. For example, in Figure 1, curve *A* has the greatest over-all reflectance and would be white. Curve *B* has less reflectance and a bluish hue, whereas curve *C* has a yellowish hue. Curve *D* has the least reflectance and would appear gray.

Curves *B* and *C* exhibit selective absorption, a property which is characteristic of a colorant.

Curves *A* and *D* are very much alike, differing only in reflectance level, but otherwise are nonselective—a property of achromatic pigments. Achromatic pigments are white, gray, or black.

Reflectance, hue, selectivity, and achromaticity are concepts of importance in this paper.

Classification of Sources of Color

We can conveniently distinguish four sources of color in raw cotton: dirt, fiber geometry or morphology, intrinsic color, and extrinsic color.

Dirt includes trash, foreign matter, and other impurities which are mechanically removable.

Fiber geometry or morphology as a source of color refers to such physical effects as due to curl, twist, cross-sectional shape, etc.

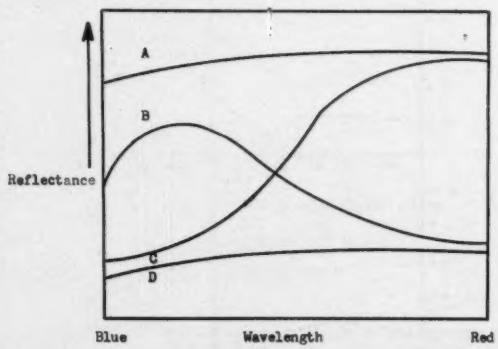

FIG. 1. *Typical spectrophotometer curves illustrating color evaluation.* A—*White.* B—*Bluish.* C—*Yellowish.* D—*Gray.*

* Presented at the spring meeting of The Fiber Society held in New Orleans, La., Apr. 24, 1953.

Intrinsic color is the inherent, genetic color of the cotton and is probably determined before the seed is planted, by the variety of the seed.

Extrinsic color is the color which is developed in the cotton by its environment or other external agencies. It cannot be removed mechanically.

The plan of this paper is to discuss the sources of color by this classification.

Dirt

Dirt is defined as dust, soil, or trash residues, which may be removed by mechanical means. The change in reflectance due to the removal of dirt in this way is, in general, not great, but is greater for the lower grades than for the higher grades. This is illustrated in Figure 2.

After bleaching, both the raw uncleaned and cleaned cottons fall on the same curve. The presence or absence of the dirt has no effect on bleachability. Dirt on the raw cotton may be a nuisance, but it is not the cause of the grayness which persists after bleaching.

Fiber Geometry

Reflectance readings might be affected by fiber geometry, or morphology—for example, the degree of convolution of the fibers. In order to determine whether or not such factors were involved, samples of the unexposed and exposed cottons, before and after bleaching, were carefully dissolved in cold concentrated sulfuric acid. The coloration of the respective solutions were noted and found to be of the same nature and proportion as in the original cotton samples. For this reason, fiber geometry is discounted as a factor of concern in this paper.

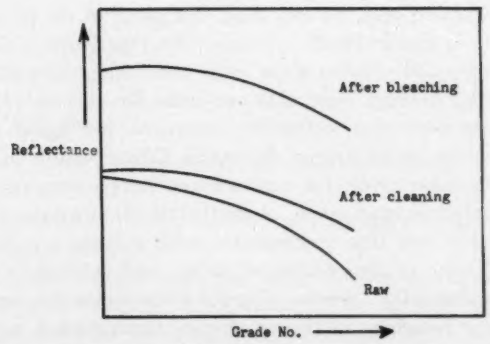

FIG. 2. *Effect of cleaning on the reflectance of cotton.*

Intrinsic Color of Cotton

The first recorded mention of the problem of color in cotton may have been made by Penot in 1836 [9]. With him there also began what has been a curiously one-sided approach to cotton bleaching. The variations in bleachability seem to have been attributed more to bleaching conditions than to variations in the cotton.

The first investigation of the nature of the pigments in cotton fiber seems to have been that of Schunck in 1868 [10]. Schunck believed the coloring matter of cotton fiber to be an amorphous mixture of two nitrogen-containing components, one soluble and the other insoluble in alcohol. The work of Schunck was quoted as late as 1921 [5], indicating that little had been done on the problem since then.

Shortly after this, two different theories about the chemical composition of the coloration in cotton began to take form. The first of these maintains that the flavones, orange-yellow pigments, are responsible. This was suggested in 1928 by Hess [2] in his book "Die Chemie der Zellulose." Flavones usually occur in the form of glucosides, and hence we have Wittmack's suggestion [12] that the flavone derivative in cotton is the glucoside.

According to the second theory, advanced in 1930 by Oparin and Rogowin [8], the coloring matter is chlorogenic acid, a compound which had been isolated earlier from the coffee bean. As a result of detailed chemical consideration of the structure of this compound, however, we are led to the conclusion that the coloring matter of raw cotton is more likely a flavone. Chlorogenic acid is, at most, a precursor of a pigment, and not a pigment itself.

Utilizing the techniques recommended in the literature for the isolation and identification of these intrinsic colorants, repeated attempts in our laboratory to isolate any of these coloring matters from bleached cotton have forced us to the conclusion that these compounds are absent. Flavones are removable from raw cotton by commercial bleaching procedures. Obviously, we must look for some other agency, rather than intrinsic colorants, as the source of the grayness persisting after bleaching.

Extrinsic Color

In order to study the effect of environment on the color of cotton, we secured cottons directly from the field, after varying periods of exposure.

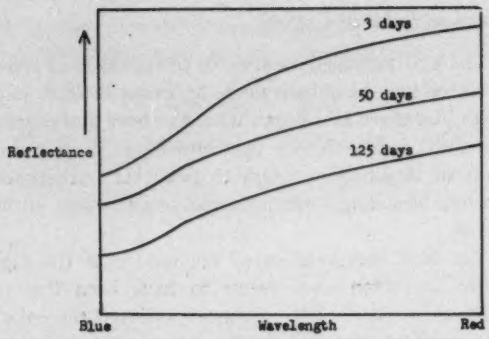

FIG. 3. *Effect of field exposure. Reflectance curves for cotton picked at intervals following opening. Before bleaching.*

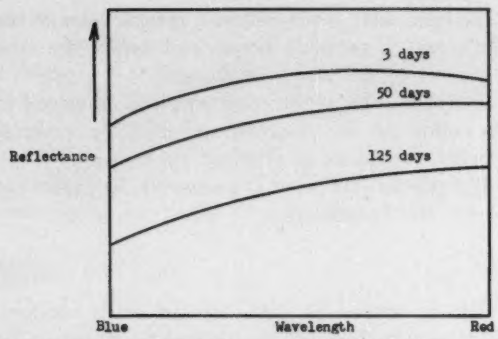

FIG. 4. *Effect of field exposure. Reflectance curves for cotton picked at intervals following opening. After bleaching.*

A field was selected from which all the cotton specimens were taken. Samples were picked after opening 3, 50, and 125 days, respectively, and were analyzed in the spectrophotometer both before and after bleaching. Reflectance diagrams for these samples are given in Figures 3 and 4.

It will be observed that the curves for all samples are essentially similar, differing only in their vertical displacement. The curves for the raw cottons exhibit selectivity as well as achromaticity. After bleaching, selectivity has been largely removed and achromaticity is now more clearly apparent.

These data are rearranged in Figure 5 to show the effect of weathering conditions on reflectance at a median wave length. Since the shapes of the curves are the same, this comparison can be made. It can be seen that increasing the exposure time reduces the reflectance of both the raw and bleached samples similarly.

The effect of weathering on cotton in the field has received much attention in the literature [1]. In particular, Nickerson [6] and Marsh [4] discussed the field exposure which results in the graying of the cotton fiber and in lower grades. It has been suggested that fungi are the source of this discoloration.

It was established by Siu [11] that these fungi first attach themselves to the surface of the cotton fibers, next penetrate the fiber walls, and then infect the lumen of the fiber, as illustrated in the idealized sketches in Figure 6. On the basis of this explanation, the extent of this penetration might be expected to be a function of the severity of the weathering conditions, and this severity, in turn, would control the extent of discoloration in cotton.

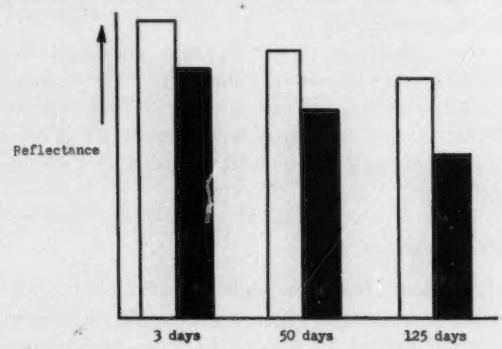

FIG. 5. *Effect of exposure on reflectance.* White—*Raw cotton.* Black—*Bleached cotton.*

We learn from Marsh [4] that three organisms are generally present whenever these weathered, dark-colored cottons are examined: *Alternaria, Cladosporium,* and *Fusarium moniliforme.* The *Alternaria* are blackish, the *Cladosporium* are dark brown, and the *Fusarium moniliforme* are near white.

Accordingly, we examined this phase of the problem in greater detail. Samples of unweathered white cotton and samples of the same cotton after 12 weeks of weathering were obtained from Dr. Marsh [4]. The weathered cotton was examined and found to contain fungi, largely *Alternaria, Cladosporium,* and *Fusarium,* while the unweathered sample was relatively free from fungi. A portion of the unweathered cotton was then impregnated with a water extract of some of the weathered cotton and subsequently incubated for 2 weeks. The color analyses before and after bleaching of the weathered, unweathered, and unweathered but incubated cottons are shown in

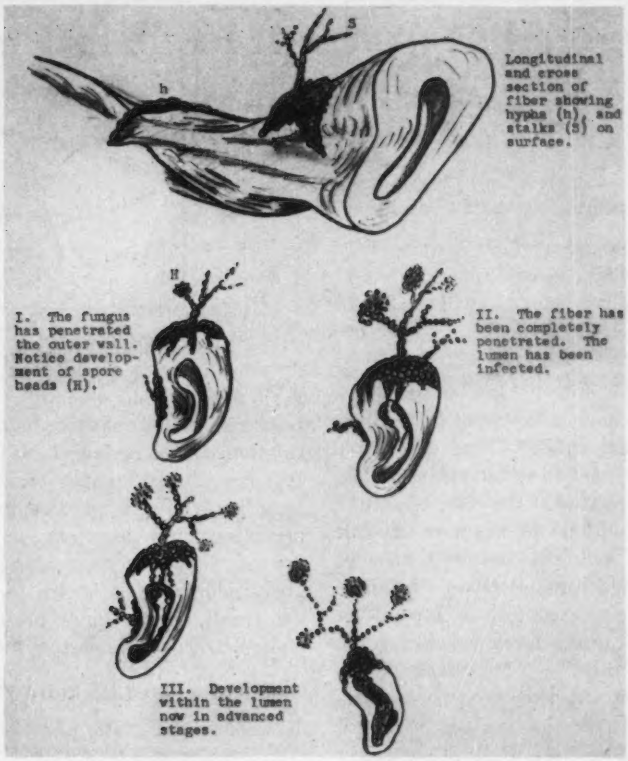
S
h
Longitudinal and cross section of fiber showing hypha (h) and stalks (S) on surface.
H
I. The fungus has penetrated the outer wall. Notice development of spore heads (H).
II. The fiber has been completely penetrated. The lumen has been infected.
III. Development within the lumen now in advanced stages.

FIG. 6. *Idealized sketches of* Cladosporium *in various stages of development on cotton.*

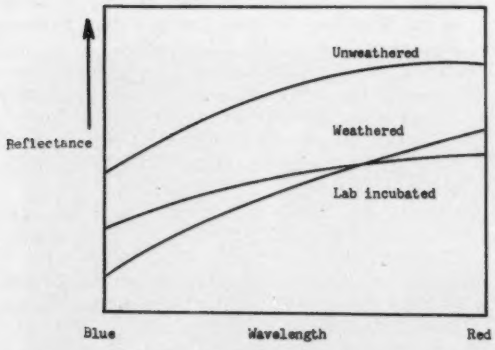

FIG. 7. *Color analysis of some raw cottons.*

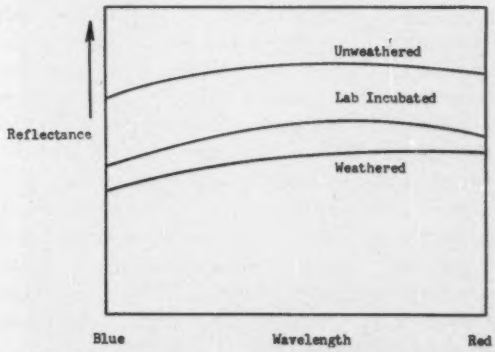

FIG. 8. *Color analysis of some bleached cottons.*

Figure 7 for the raw samples and in Figure 8 for the bleached samples.

These samples show that the laboratory incubation caused a reduction in reflectance of the unweathered sample which persisted after bleaching. In the raw samples, the laboratory-incubated sample was distinctly yellower in hue than the weathered sample, as evidenced by the skewness of the two curves. The bleached samples, on the other hand, did not show this skewness, which thus is further support for the theory that some of the intrinsic color may be lost during weathering. The intrinsic color completely disappears, however, upon bleaching.

We recognize that the samples tested in this

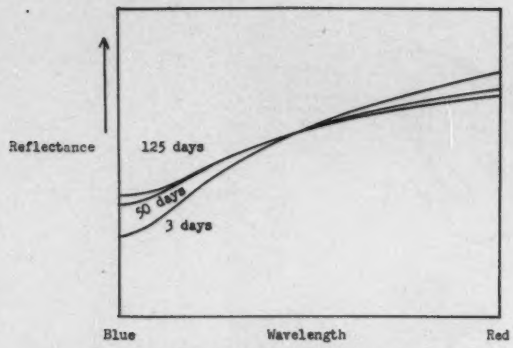

FIG. 9. *Analytical curves of Figure 3 (raw cottons) superimposed.*

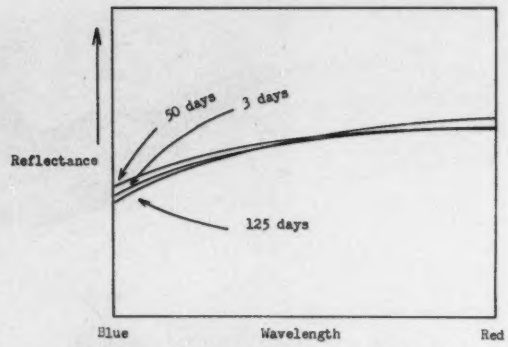

FIG. 10. *Analytical curves of Figure 4 (bleached cottons) superimposed.*

exposure series might have differences in intrinsic color as well as extrinsic color. These differences would appear in the form of hue variations, especially with respect to the yellow side of the color spectrum. Because of the bleachability of the flavones, any hue differences existing in this raw cotton should be eliminated in the bleached cotton—unless, of course, the flavones had been rendered fast as a result of combinations or degenerations developed during the course of exposure and time.

This was confirmed by superimposing the analytical curves of the exposed cottons, raw and bleached, at their respective mid-points. This is shown in Figures 9 and 10.

It can be noted that in Figure 7, the curves of the exposed raw cottons are skewed or selective with respect to the white raw cotton, but in Figure 8 the curves for all the bleached samples are substantially congruous. This indicates that the hue factor is substantially eliminated by bleaching and confirms the bleachability of the intrinsic color. Furthermore, the condition of achromaticity is again established.

Referring to the curves again, the raw white cotton is skewed to show that it contains more yellow than dull or "blue" cotton. This suggests that a white cotton in the process of becoming a "blue" cotton as a result of exposure and weathering will also lose some of its yellowness.

Summary and Conclusions

The color of cotton has been defined, and the sources of color in cotton have been classified. A review of past work on the nature of the intrinsic color of cotton is given, and the chlorogenic acid theory of the source of unbleachable color is criticized. It is concluded that the intrinsic colorants of cotton should not survive bleaching.

The literature reporting on the sources of extrinsic colorants is also reviewed. It is shown that the color remaining in cotton after bleaching is of this nature. It is finally shown and demonstrated that the color persisting after bleaching is due to fungi which flourish under the correct weather conditions. The undesirable color is shown to be achromatic and is the result of the fungi presence itself. Spectrophotometric data supporting the analysis are given.

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(Manuscript received September 9, 1953.)

Structural Details of the Surface Layers of Wool

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Introduction

Studies by Lindberg, Philip, and Gralén [7] revealed that the wool fiber is covered by a thin membrane, the epicuticle, with a thickness of an order of magnitude of 100 Å. This membrane can be separated from the fiber by different chemical methods. Elementary analyses of these membranes give different chemical compositions for the different methods employed [5]. Similar membranes can be separated from other structures of animal origin [6, 15, 16].

From studies of the formation of the Allwörden blister, Lindberg [8] concluded that the epicuticle forms a continuous skin over the whole fiber. A review of the role of this membrane was given by Lindberg, Mercer, Philip, and Gralén [9]. However, doubt has been aroused as to the validity of the conclusion of the continuous skin and the sufficiency of the underlying experimental evidence. Zahn [19], for instance, stated that every scale is surrounded by a thin membrane, and O'Reilly *et al.* [12] made a similar suggestion.

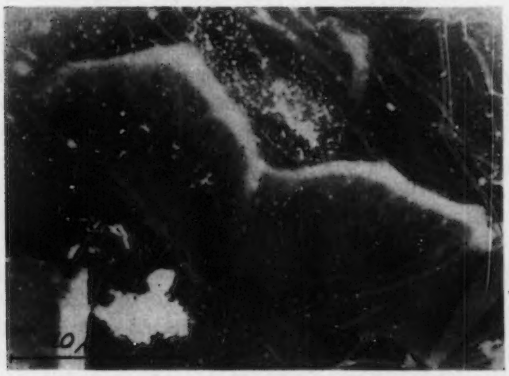

FIG. 1. *The exocuticle layer becomes thinner with increasing distance from the scale edges. The edge in the middle, common to the two scales, likewise becomes thinner; finally the surface layers from both scales end up in a common layer of epicuticle. Phenol-tryptic digested specimen, deposited on SiO, unshadowed.*

Phenol-Tryptic Digestion

Treatment with phenol at 100° C for 2 hrs. followed by digestion with trypsin at pH 8–9 gave specimens in which the scaly structure still could be recognized [10]. These studies have been continued.

Figure 1 is from a preparation obtained by this method. The specimen is unshadowed and deposited on a SiO membrane. The scale edges are rather thick, but the exocuticle layer adhering to the part next to them gets thinner with increasing distance from the edges. The edge in the middle, common to the two scales forming the specimen, likewise gets gradually thinner, and finally both scales end up in a common sheet of epicuticle, free from contaminating substance.

FIG. 2. *The ends of a scale edge end blindly in a sheet of epicuticle and adhering exocuticle. The streakinesss, with a periodicity of approximately 0.3μ, runs continuously from the one scale to the other. Phenol-tryptic digested specimen, Pd-shadowed at an angle 4:1.*

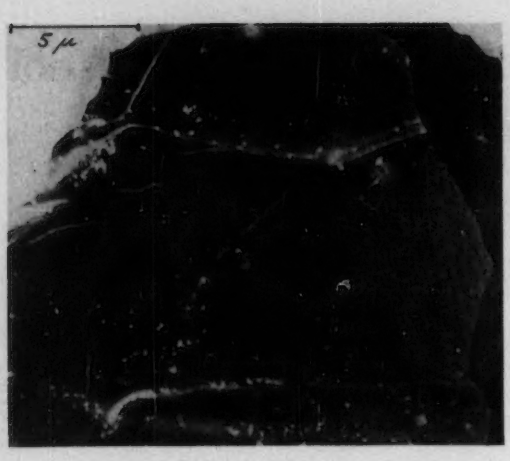

FIG. 3. *Ridges (curled or corkscrew-shaped structures running parallel to the main direction of the scale edges) are seen. One of them protrudes from the scale edge and seems to be reinforcing it. A string runs between two scale edges. The streakiness runs in the length direction of the fiber. Phenol-tryptic digested specimen, deposited on SiO, unshadowed.*

FIG. 4. *Striated surface-layer sheet with strings and the curled remnants of a ridge. The holes are located on the strings. Phenol-tryptic digested specimen, Pd-shadowed at an angle 4:1.*

Figure 2 shows part of a specimen from the same type of preparation, but which is Pd-shadowed at an angle 4:1. In the middle of the picture a scale edge

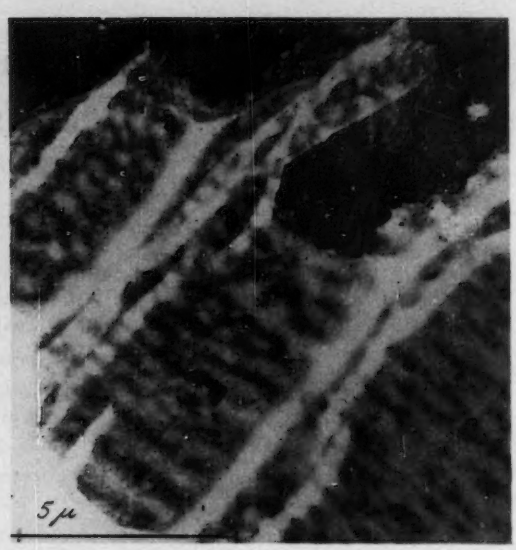

FIG. 5. *Phenol-tryptic digested specimen in an early stage of degradation. The streakiness has a periodicity of about 0.6μ. Pd-shadowed at an angle 4:1.*

can be seen, the ends of which end blindly in a sheet built from epicuticle and adhering exocuticle. This sheet is striated, and the repeating unit in the streakiness is approximately 0.3μ. In the area where the scale edge is broken, the streakiness runs continuously from the one scale to the other without being affected by the former presence of the scale edge.

Among the details observable on Figure 2 is a thin string running from the broken scale edge to another edge. A similar structure is more distinctly visible in Figure 3; this specimen originates from the same preparation, but is unshadowed and deposited on a SiO membrane. This string structure, which runs from the scale edge in the upper part of the figure, obscures the uniform streakiness and has an appearance similar to that of a welding-joint. It can be seen that the scale edge is double and that a slightly curled structure protrudes from this edge. Two more of these curled structures are also visible, running parallel with the first one and with the main direction; these structures are of the same type as the ridges mentioned by Gralén [1] and by Lagermalm [4]. The term "ridge" refers to this type of structure, whereas the word "string" refers to the structure shaped like a welding-joint.

Figure 4 shows strings and the curled remnants of a ridge. As can be observed, the holes in the sheet

FIG. 6. *In the thicker area near the scale edge the fine streakiness is replaced by a streakiness of the higher repeating unit. Phenol-tryptic digested specimen, deposited on SiO, unshadowed.*

are located on the strings, which is a phenomenon generally observed for this type of specimen.

The specimens give a somewhat different picture of the ridges and the streakiness when the digestion is interrupted at an earlier stage. Figure 5, which is of a specimen Pd-shadowed at an angle 4:1, shows a streakiness where the repeating unit is approximately 0.6μ and where the thicker part of the streakiness is about 500 Å thick and the thinner part about 200 Å thick; the average thickness of the common and more completely digested type of specimens obtained by this method is about 100 Å. The ridges on Figure 5 are also of a much more solid type than the usual type.

Figure 6, which is unshadowed, shows how the fine streakiness in the thicker area near the scale edge is replaced by the streakiness with the higher repeating unit.

A general view of a larger surface-layer sheet is shown in Figure 7, which was compiled from several detail exposures. Pure epicuticle, areas thickened by exocuticle, scale edges, and ridges are seen. The specimen is shadowed with Pd at an angle 4:1.

Figure 8, which is unshadowed, shows a similar piece. Even the strings are easily visible here. In some cases they run as continuations of the scale edge, being much thinner and more distinct than the scale edge itself.

Several of the structural details discussed can be seen if wool fibers are broken down by treatment with molten urea at about 135°C for 5–10 min., followed by tryptic digestion.

Alkaline Digestion

The method first used for separation of the epicuticle from the fiber was the Na_2S method [7]. This method did not reveal much of the structural details of the surface layers except the existence of an alkali-resistant membrane with an adhering cementing substance and the perforation and starting degradation brought about by the alkali [3, 14]. When preparing specimens by the alkali method, it was found, however, that by interrupting the treatment at an early stage, results could be obtained bearing striking similarity to the results from the phenol-tryptic experiments. Figure 9 is from a specimen obtained by treating wool with 1% Na_2S solution at room temperature for 5 days and afterwards for 1 day with a solution saturated with urea at 50° C; the specimen is Pd-shadowed at an angle 4:1. This aftertreatment does not, however, seem to be of significant importance, since similar results were also obtained by alkali treatment alone. A string structure which has been branched somewhere in the area of the hole in the sheet is seen. It should be noted that part of this string has been loosened from its base and turned about 180° from its original position, which was probably on one border line of the hole. This shows that the string cannot be a fold, but is a real structure. The periodicity of the streakiness is about 0.3μ, and it is obscured by the string. Thus, both of these factors have the same appearance as on the phenol-tryptic treated specimens.

The string type of structure also appears in other modifications. One is shown in Figure 10, which is from the same preparation as the previous one and shadowed in the same way. Here the string has more of a welding-joint appearance than in Figure 9. It breaks the regular pattern of streakiness and is surrounded by an area with dots. Dots are also found in the neighboring area, but their concentration is highest near the string. Another feature in this picture is that near the peak of the peninsula-shaped sheet there is a piece having the appearance

of the conventional Na_2S-produced epicuticle with no other structural details than a starting disintegration of the membrane. This area is separated from the rest of the sheet by a string similar to that in Figure 9.

A string similar to the curved one on Figure 10 but still thinner is seen in Figure 11. The string is surrounded by dots in the same way, and in some places it is so thin and so intimately joined to the background that it can hardly be distinguished from it. The streakiness is not as pronounced as in Figure 10, thus indicating a lower specimen thickness in Figure 11.

Still another modification of structure separating two fields is seen in Figure 12,* which is from the same preparation as Figures 9–11. The two striated

* The black, parallel lines in this and some of the other pictures are due to defects in the plates.

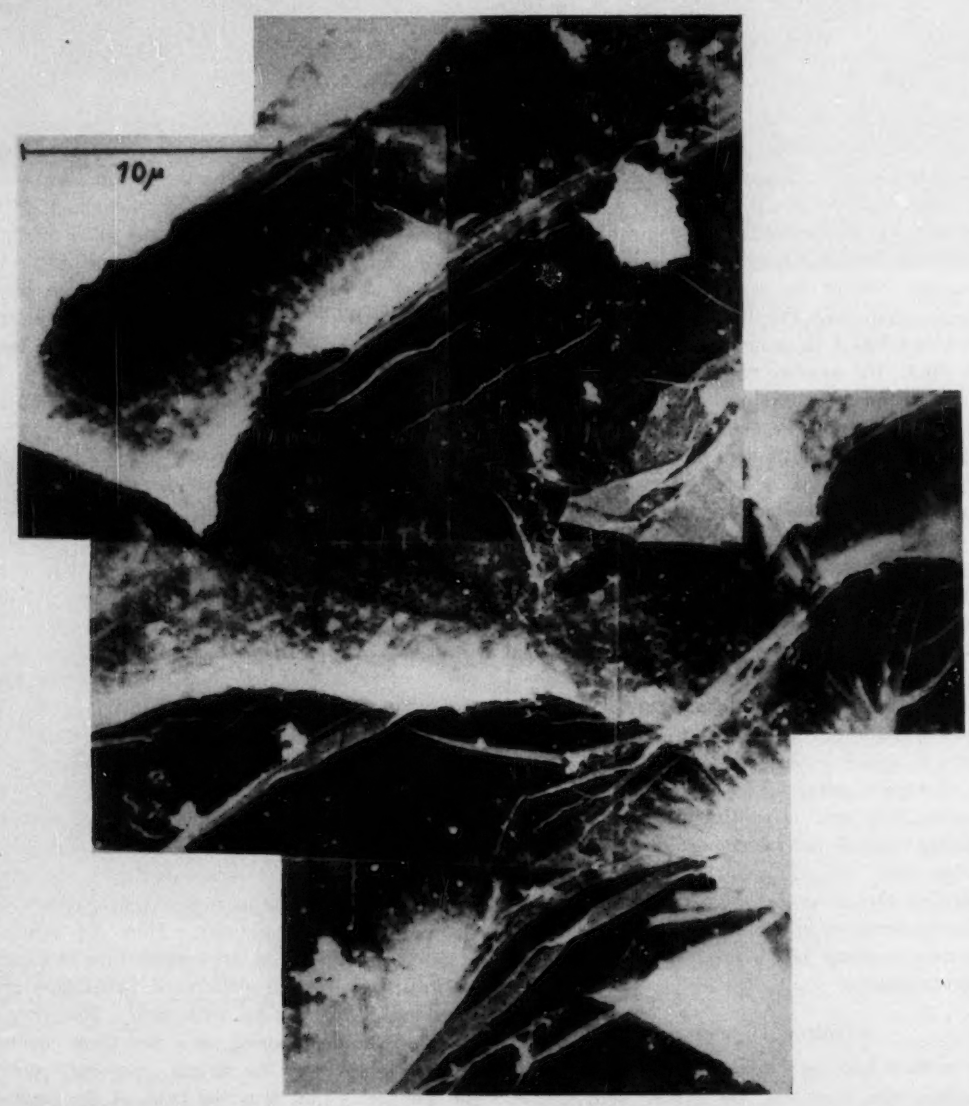

FIG. 7. *General view compiled from several detail exposures. Pure epicuticle, areas thickened by exocuticle, scale edges, and ridges are seen. Phenol-tryptic digested specimen, Pd-shadowed at an angle 4:1.*

fields are here separated by a narrow area where the sheet is somewhat thinner than elsewhere. Small pieces of epicuticle in a stage of starting disintegration are separated from the main parts of the sheet in a manner similar to that shown in Figure 10.

Figures 13 and 14 are from a specimen obtained by treating the wool with dilute Na_2S solution for 3 days at room temperature. This specimen was not treated with urea solution. It is unshadowed. Figure 14 is a higher magnification of a detail from Figure 13 plus the neighboring area. It can be seen how the scale edges are very often built up by double lines. These lines do not always end with the scale edge, but run individually and independently of each other into the areas of other scales. They do not even run strictly parallel along the scale edges.

Comparison with Brominated Specimens

Epicuticle separated from wool by chlorination or bromination is in most cases structureless. Schuringa and Algera [17], however, reported the occurrence of a small percentage of striated membranes. The observation has been confirmed by experiments in this laboratory. Sometimes, however, other structures may also be observed, such as the dotted lines on gold-shadowed specimens from bromated wool, as reported by Gralén [1] and Lagermalm [4]. The dots seen in these cases may be due to the aggregation of the gold used for shadow-casting, caused by the heat evolved by the absorption of electrons. More heat will be evolved in the thicker parts of the specimen, such as scale edges, strings, and ridges.

FIG. 8. *Strings run as continuations of the upper or lower limitations of the scale edges. The strings are thinner than the scale edges. Phenoltryptic digested specimen, deposited on SiO, unshadowed.*

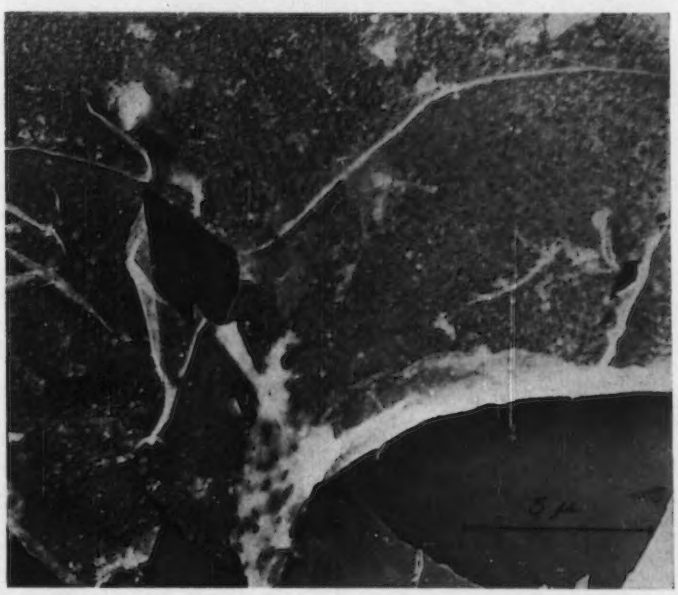

FIG. 9. *The strings branch from a point somewhere in the area of the hole in the sheet. Part of one string has been loosened from its base and turned about 180° from its original position. Streakiness, with a periodicity of about 0.3μ, covers most of the sheet. Alkali-digested specimen, Pd-shadowed at an angle 4:1.*

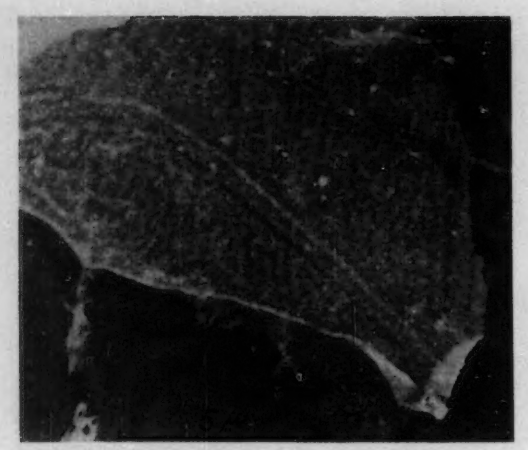

FIG. 10. *Welding-joint type of string breaks the regular pattern of streakiness. A piece of epicuticle in a stage of starting disintegration is separated from the main part of the sheet by a somewhat thicker string. Alkali-digested specimen, Pd-shadowed at an angle 4:1.*

FIG. 11. *The string is so thin that in some places it can hardly be distinguished from the epicuticle itself. Alkali-digested specimen, Pd-shadowed at an angle 4:1.*

In some cases the dots form distinct lines; in other cases the dots are spread almost uniformly over the greatest part of the sheet, and the lines occur more like diffuse, white bands. It seems probable that these lines are related to the structures observed on preparations made according to other methods.

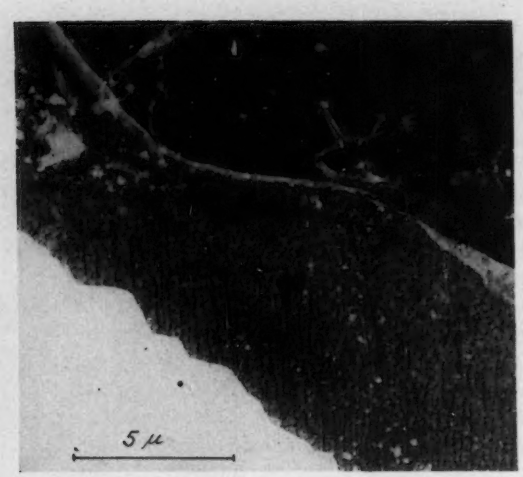

FIG. 12. *Two striated fields are separated by a narrow area where the sheet is somewhat thinner than elsewhere. This narrow band is probably due to a string's having been torn away from its original position. Disintegration epicuticle is seen near the borders of the sheet. Alkali-digested specimen, Pd-shadowed at an angle 4:1.*

Discussion and Conclusions

The part of the surface layers considered in this paper is composed of the epicuticle and the structural details underneath it, which are not merely remnants of loosely attached scales. This is demonstrated not only by the thickness of the specimens, but also by the fact that in several cases the layers are connected with or gradually change into areas of pure epicuticle or areas in a state of beginning disintegration of the same type as reported in earlier publications [1, 3, 4, 14].

Although methods of separating the surface layers from the rest of the fiber vary in their way of action and give specimens differing in their chemical reactivity and composition, nevertheless they reveal similar or even identical details in the structure of the surface layers.

It is evident that the extension of the epicuticle is not restricted to the single scale. A continuous sheet of epicuticle covers several scales, and perhaps the whole fiber. This was earlier reported by Gralén and Lagermalm [2]. Mercer and Golden [11] recently published evidence from light optical studies of blisters formed by oxidation followed by treatment at pH about 8. Parisot and Leveau [13], however, showed that the walls of these blisters consist of a thick epicuticle. Blisters covering several scales

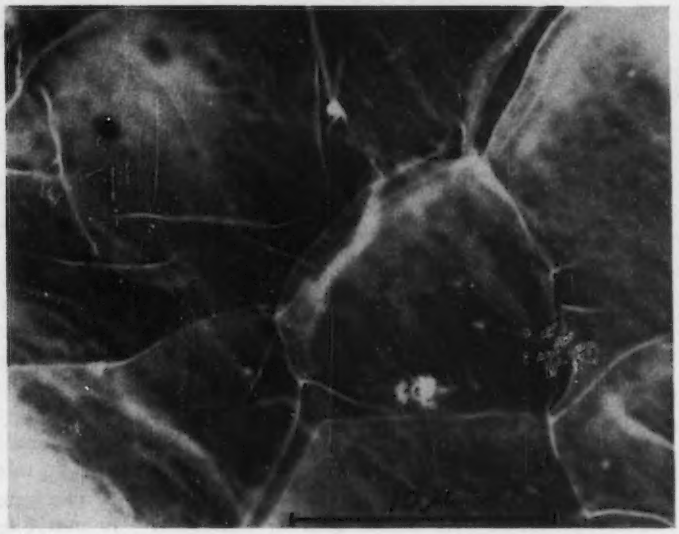

FIG. 13

FIG. 14

FIGS. 13 AND 14. *The scale edges are outlined by strings. It can be seen how some of the edges are formed by two strings, each of them following their individual path and in many cases forming part of several edges. Alkali-digested specimen, deposited on SiO, unshadowed.*

were also observed by Sörvik [18]. The suggestion made by Zahn [19] that the extension of the epicuticle should be restricted to the single scales and the similar suggestion by O'Reilly *et al.* [12] thus do not hold.

It is in some cases a matter of definition whether the structural details discussed above are referred to the epicuticle or to the exocuticle. This is particularly true for some of the string type of structures, which can be so intimately connected with the epicuticle that they can hardly be distinguished from it. However, we may, by definition, refer these structures to the exocuticle.

The scale edges are characterized by thick deposits of exocuticle substance. Sometimes double lines can be recognized at the scale edges. The width of the

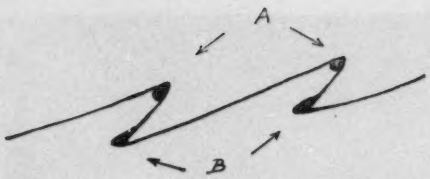

FIG. 15. *When scale edges are outlined by two strings, these strings generally correspond to the position* A *and* B. *Between* A *and* B *ordinary exocuticle substance is deposited, thus forming the thick scale edge.*

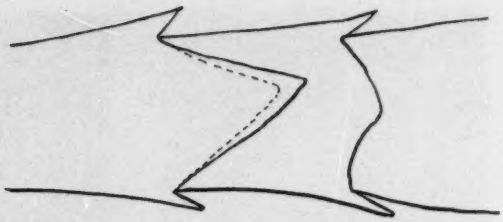

FIG. 16. *The degree of overlapping may vary along the scale edge.*

deposits, or the distance between the double lines, is about 0.5–1.0μ, which indicates that this double structure originates from the slight overlapping at the scale edges, visible in the light microscope. In some cases ridges follow the scale edges, where they may have a reinforcing effect. The ridges, which have a corkscrew-shaped structure, run parallel to the main direction of the scale edges. The common exocuticle substance, without structure or with streakiness, is thickest near the scale edges and gradually gets thinner with increasing distance from the edges.

The string structures observed are of different types. Some of them may represent different degrees of degradation of the same basic structure. From the material collected it seems very probable that the strings and the scale edges are related in such a way that the edges often are constructed according to a basic pattern of two strings running more or less parallel along the edge. These strings correspond to the upper and lower limitations of the scale (*A* and *B* in Figure 15), visible in the light microscope.

Between *A* and *B* ordinary exocuticle substance is deposited, thus forming the thick scale edges often visible. The overlapping is not necessarily the same along the length of the edge (Figure 16), and it is also possible that the difference in *niveau* between the upper and lower scales varies along the edge.

The strings are not restricted in length to that of a single scale edge, but run into the areas of other scales and may take part in the formation of other scale edges. These strings do, however, run through areas where they do not form edges. Strings like those in Figures 9, 10, and 11 are probably of this type. The strings may be regarded as reinforcing elements, the epicuticle being fastened along them to the exocuticle and perhaps even to the endocuticle in a more intimate union than are the areas constituting the main part of the epicuticle. If part of the epicuticle has been loosened, it may still be sticking to underlying layers by means of the fixation along the strings. If we try to accomplish the separation of the epicuticle from the bulk of the fiber by mechanical means, such as shaking or ultrasonics treatment, the surface-layer sheet may preferentially be broken or ruptured along these strings. This may explain why the holes are so often found at the strings and not in the much larger areas of the sheet.

If we take the pictures from the Na_2S treatment as an example, we might suppose that Figures 9, 10 and 11 represent different steps of degradation of the strings in the order mentioned. In Figures 10 and 11 this is quite evident. The break in the regular pattern of streakiness in Figure 12 may be due to the string in some way being torn away from the sheet.

It is obvious that the material building up the strings does not have the resistance of the epicuticle itself. This is evidenced by the fact that the strings are not found in preparations obtained by long-time treatment with Na_2S solution and are very seldom found in preparations obtained by chlorination or bromination. The material is most probably of an ordinary protein type.

The fixation of the epicuticle to the scale edges, observable during the formation of the Allwörden blisters, is not due to a fixation of the epicuticle between two overlapping scales, as in a cloth-peg. It is obtained by the intimate fixation to the underlying layers caused by one, or in many cases two, strings, and also by a gluing effect of a thicker or denser layer of exocuticle material at the edges. The effect observed by Mercer and Golden [11] and by Sörvik [18] of blisters covering several scales indicates that these strings are very sensitive to oxidation followed by mild alkaline treatment or to subsequent oxidation and reduction treatments.

The ridges may have a function similar to that of the strings in contributing to the fixation of the layers relative to each other. In some cases, as when reinforcing the scale edges, the task of the ridges is also similar to that of the strings. Ridges are seldom observed on urea-treated specimens. This indicates

FIG. 17. *Two types of streakiness are recognized in the exocuticle, both running in the length direction of the fiber. The one, lying nearer the epicuticle, has a repeating unit of about 0.3μ, whereas the other, lying nearer the endocuticle, has a repeating unit of 0.6μ. The latter is also the more easily digested of the two.*

a difference in reactivity or chemical composition between strings and ridges, the latter being more easily extracted than the former.

The phenol-tryptic digested specimens which have not been fully broken down but still have a thickness of about 200 Å. measured at their thinnest parts show a thicker type of ridge than the type found in thin specimens. In this case it is difficult to distinguish between ridges and strings because the adhering ordinary exocuticle substance obscures the structures.

On thin specimens it is often difficult to distinguish between ridges and folds. In many cases folds are found in connection with the ridge structure, and ridges may be seen as superimpositions on the folds.

The streakiness is visible even when the specimens are digested to a degree where only a thin, rather uniform exocuticle layer is left in contact with the epicuticle. A repeating unit of about 0.3μ seems to exist in these cases, whereas thicker deposits of a somewhat higher repeating unit, about 0.6μ, are observed in specimens digested to a lesser degree.

Schematically the arrangement of the streakiness may be as depicted in Figure 17, the repeating units of higher order lying nearer the endocuticle and being digested more easily than the other part.

The exocuticle is thus a layer containing several structural details, and is not merely a layer of unordered cementing substance. Besides the cementing function, it seems to have a role in the modeling of the fiber surface, covering possible unevenness in the surface of the endocuticle and thus offering a relatively smooth surface to be covered by the epicuticle.

Summary

Electron microscopical studies have been made on the surface layers of wool, separated from the rest of the fiber by several methods. Despite the variations in chemical composition obtained by the different methods, details of the same basic structure are revealed. The epicuticle covers the fiber as a uniform layer, without being limited to the individual scales. The exocuticle comprises several structural details: ridges, wavy structures running parallel to the scale edges; strings running between the scale edges and forming the top and bottom limitations of the edge at the overlappings; streakiness in the length direction of the fiber with a fine periodicity of about 0.3μ and a coarse periodicity of about 0.6μ. A thickened layer of exocuticle is deposited between the ridges at the scale edges. The thickness of the exocuticle layer decreases with increasing distances from the scale edges.

Acknowledgment

The author would like to express his sincere thanks to the head of the Institute, Professor Nils Gralén, for stimulating discussions and friendly criticism. He is indebted to the International Wool Secretariat for a research fellowship, enabling him to work with these problems.

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Preparation of Microbiologically Resistant Wool by Means of Chemical Modification

Part II: Paper Chromatographic Investigation of the Samples*

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IN Part I Zahn and Wilhelm [5] gave a summary of methods which are suitable for protecting wool against rotting in the soil-burial test by means of chemical modification. The individual chemically modified wool samples were characterized chemically by the cystine content and the acid and alkali solubilities. It was deemed desirable to employ the paper chromatographic method for establishing possible changes in the amino acid composition of hydrolysis products of the samples. Results by this method may, of course, only be expected if the bond resulting from the chemically modifying reaction is capable of withstanding hydrolysis of the wool with 6*N* HCl. On the other hand, it was found that samples which yield hydrolysis products incapable of withstanding attack by 6*N* HCl nevertheless often give rise to anomalous deviations in amino acid composition so that it seemed advisable to examine all samples systematically by the paper chromatographic method.

* Part I appeared in the September, 1953, issue of TEXTILE RESEARCH JOURNAL.

Part II, with a full description of chromatographic techniques, will be published in German in *Melliand Textilberichte*, February, 1954.

Results

Wool Yarn Modified with Monofunctional Compounds

Wool which had been treated with epichlorohydrin and then hydrolyzed showed the following deviations from untreated wool on the paper chromatogram: The spot for lysine disappeared. This also applied to the hydrolysis product of the residue which was insoluble in 4*N* HCl at 65° C. The histidine spot on the coupled chromatogram was very weak. Cysteic acid made its appearance as an additional product. On the two-dimensional paper chromatogram of the hydrolysis product of acetylated wool, a spot which reacts with ninhydrin was located between cystine and lysine (see Figure 1). The constitution of this spot was not established. It exhibited an R_{lys} value of 0.79 † on the continuous running chromatogram (see Figure 2). For the hydrolysis product of dinitrophenylated wool, the tyrosine spot was absent and

† This signifies that the compound in question migrated 79% as much as lysine.

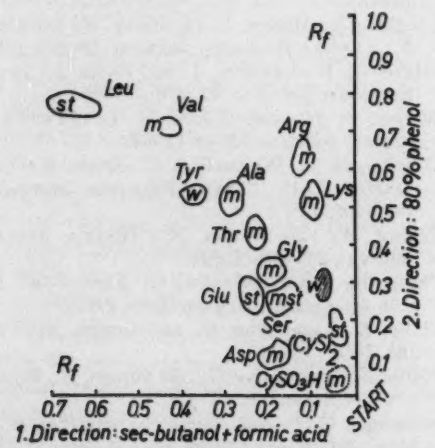


FIG. 1. *Two-dimensional paper chromatogram of the hydrolyzate of wool boiled with acetic anhydride (Schleicher Schüll 2043b, ascending technique).*

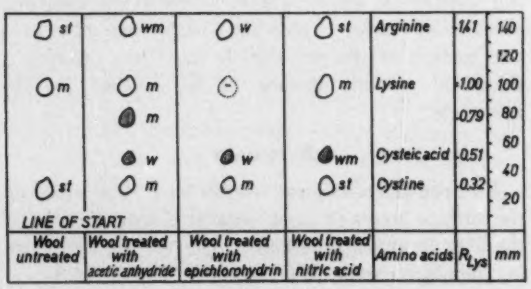


FIG. 2. *One-dimensional continuous paper chromatogram (descending technique, 6 days, Whatman No. 1, solvent* sec*-butanol-formic acid-water).*

the lysine spot was considerably diminished in intensity. In the neighborhood of the leucine group were found the yellow spot from ε-dinitrophenyllysine and the colorless spot from *o*-dinitrophenyltyrosine, which reacts with ninhydrin (see Figure 3). The weak histidine spot was apparent in the coupled chromatogram. For the hydrolysis product of nitrated wool, the tyrosine spot was missing and the presence of cysteic acid could be demonstrated on the continuous running chromatogram.

Wool Treated with Bifunctional Compounds

For the hydrolysis product of formalized wool, the tyrosine spot was absent. For wool which had been treated with Kaurit 140,* the tyrosine spot was very weak. The same applied to wool which had been treated with butanediol-*bis*-chloromethyl ether.†

Paper chromatograms of wool samples treated with *bis*-chloromethyldimethylbenzene in acetone-bicarbonate solution were quite striking, as were also those for wool treated with acetone and bicarbonate alone. A clear lanthionine spot made its appearance here. The hydrolysis product of wool treated with *bis*-chloromethyldimethylbenzene no longer contained lysine.

* B.A.S.F. (Badische Anilin- und Sodafabrik), Ludwigshafen, Germany.

† Farbwerke Höchst, Frankfurt-Main-Höchst, Germany.

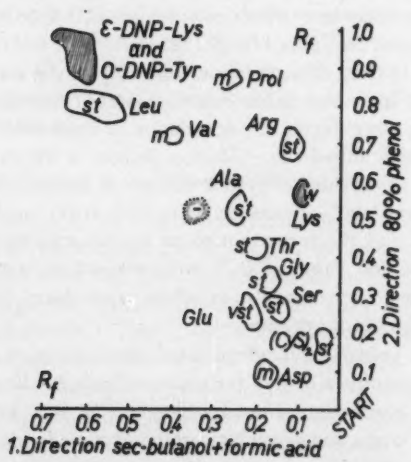


FIG. 3. *Two-dimensional paper chromatogram of the hydrolyzate of dinitrophenylated wool (Schleicher Schüll No. 2043b, ascending technique).*

Reactions at the Disulfide Bonds in the Wool Cystine Residue

For the hydrolysis product of so-called "CH_3S-wool," which had been reduced with thioglycolate and subsequently methylated with methyl iodide, the S-methylcysteine spot was to be found approximately between valine and alanine on the paper chromatogram (see Figure 4).

Wool which had been treated with 1,2-dibromoethane after reduction and then hydrolyzed exhibited on the paper chromatogram the S,S'-ethylene-*bis*-

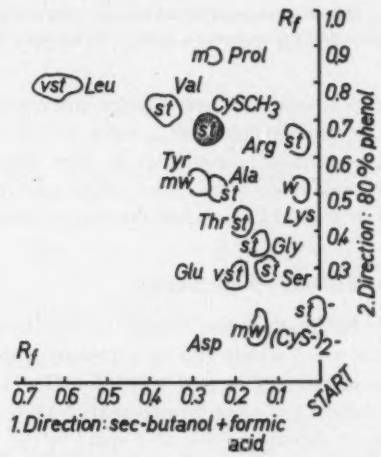


FIG. 4. *Two-dimensional paper chromatogram of the hydrolyzate of reduced and methylated wool (Schleicher Schüll No. 2043b, ascending technique).*

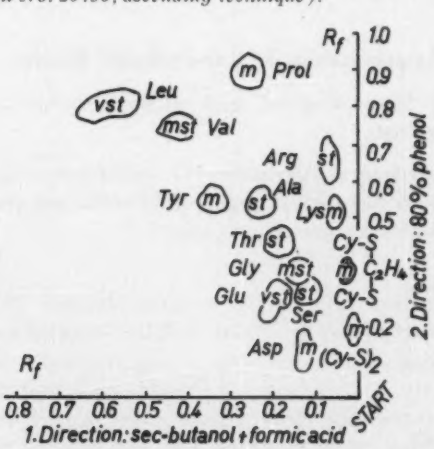


FIG. 5. *Two-dimensional paper chromatogram of the hydrolyzate of wool reduced and treated with ethylene dibromide (Schleicher Schüll No. 2043b, ascending technique).*

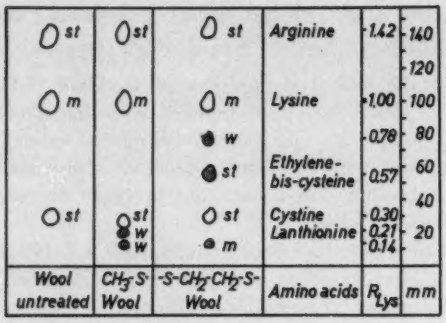


FIG. 6. *One-dimensional continuous paper chromatogram (descending technique, 6 days, Whatman No. 1).*

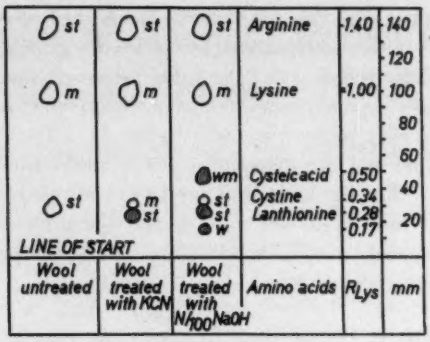


FIG. 7. *One-dimensional continuous paper chromatogram (descending technique, 6 days, Whatman No. 1).*

cysteine spot, located between lysine and cystine (see Figure 5) and having an R_{lys} value of 0.57 on the continuous running chromatogram (see Figure 6). Further weak spots of unknown origin and R_{lys} values of 0.78 and 0.14 also made their appearance here.

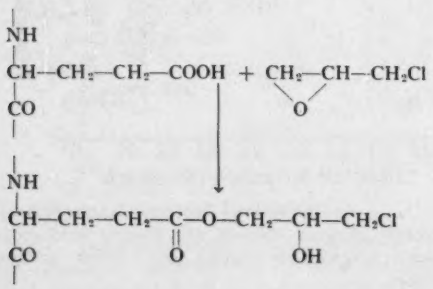
Various Methods of Treatment

Much lanthionine was found in the hydrolysis product of wool which had been treated with KCN and also in samples treated with NaOH. The hydrolysis product of wool treated with $N/100$ NaOH gave rise to definite cysteic acid spots in the continuous running chromatogram (see Figure 7) as well as to a spot migrating even slower than lanthionine ($R_{lys} = 0.17$).

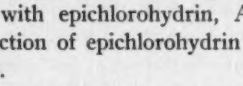
Interpretation of Experimental Results

Wool Yarn Modified with Monofunctional Compounds

According to Alexander [1], epichlorohydrin reacts with the carboxyl groups of the acidic side-chains of proteins to form ester groups:



Furthermore, the amino groups react to give



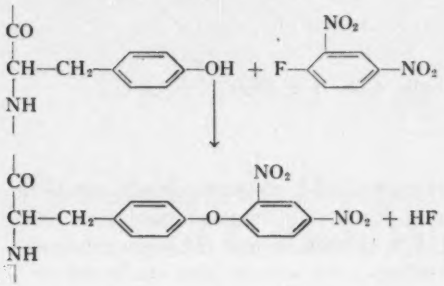
A new epoxide may now be formed by splitting out HCl from these reaction products *in situ*. As a result of his investigations on the supercontraction of wool treated with epichlorohydrin, Alexander assumed the reaction of epichlorohydrin with wool to be bifunctional.

The fact that the hydrolyzate of wool treated with epichlorohydrin does not contain lysine may be due to the *N*-alkylation reaction. The appearance of cysteic acid, however, is incomprehensible on the basis of the proposed reaction mechanism. A definite decrease in cystine content from 11.5% to 8.1%, was noted in Table III of Part I.

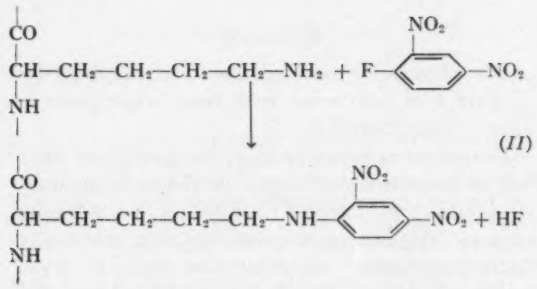
Apart from *N*-acetylation, probably of the ε-amino groups in lysine, other reactions would also appear to take place during the acetylation of wool with boiling acetic anhydride. Thus, a fission of the cystine bonds (a decrease of cystine content of approximately 10% and the appearance of cysteic acid) probably occurs and the formation of an amino acid showing a migration value of 0.79 with respect to lysine is observed. Presumably, ornithine is produced by the degradation of arginine.

The yellow dinitrophenylated wool contains only small quantities of free tyrosine and lysine. As a result of work carried out by Sanger [3], it is known that primary and secondary amino groups, imidazolyl groups, and phenol groups present in proteins react with 2,4-dinitrofluorobenzene to liberate HF. Since the number of α-amino groups at the ends of protein

chains is small, the main reactions on dinitrophenylation of wool consists in the etherification of the phenolic tyrosine groups (*I*) and the formation of ε-dinitrophenyllysine (*II*) (see [6]).



(*I*)



(*II*)

As far as the nitrated wool described in this group is concerned, the complete disappearance of the tyrosine spot is easy to understand since the tyrosine residues in the wool, as in silk, react readily with HNO_2 at positions 3 and 5 to yield 3-nitrotyrosine and 3,5-dinitrotyrosine residues. It is somewhat remarkable that, under the conditions chosen, the cystine bonds are attacked to only a very slight degree (appearance of cysteic acid).

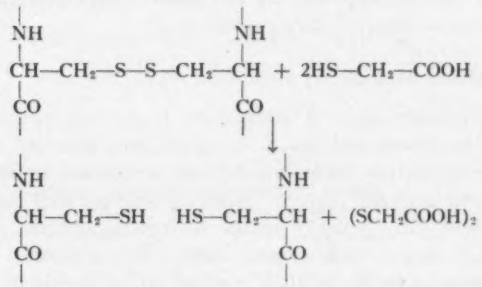
Wool Treated with Bifunctional Compounds

When it is no longer possible to detect the presence of tyrosine in the hydrolysis product of formalized wool, this does not necessarily imply that all the tyrosine residues reacted completely with the formaldehyde. Thus, the Millon reaction on unhydrolyzed, formalized wool is still positive. It is therefore presumed that the tyrosine is not destroyed until hydrolysis, at which stage it disappears in the course of secondary condensation reactions.

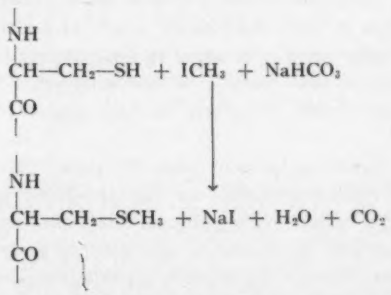
The paper chromatographic detection of lanthionine in wool treated with 4,6-*bis*-(chloromethyl)-1,3-dimethylbenzene, as also in wool treated with acetone and bicarbonate alone, explains the disappearance of approximately half of the cystine bonds present in untreated wool. The copious formation of lanthionine from wool cystine, even on warming the wool to 65°C with acetone and bicarbonate, represents the mildest process yet found for converting cystine into lanthionine. An interpretation of this reaction is not possible at the present time.

Reactions at the Disulfide Bonds in the Wool Cystine Residue

On reducing the wool with calcium thioglycolate, free sulfhydryl groups are formed from the disulfide bonds:

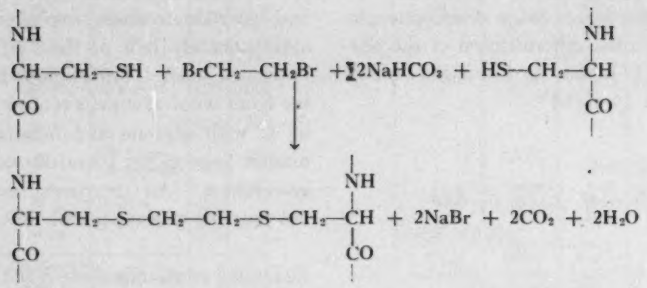


The action of methyl iodide on the HS-wool results in S-methylation, according to the following equation:



The S-methylcysteine residue is liberated on hydrolysis, and may then be readily detected on the paper chromatogram, as was demonstrated by Consden, Gordon, and Martin [2] in 1946.

On treating the reduced wool (HS-wool) with 1,2-dibromoethane, ethylene-*bis*-cysteine bonds are formed in the wool, thus giving rise to so-called "—S—CH_2—CH_2—S—wool." This reaction may be formulated in the following manner:



S,S'-ethylene-*bis*-cysteine was prepared by Wollemann from wool chemically modified by the Harris process and identified with the synthetic compound [4]. The appearance of S,S'-ethylene-*bis*-cysteine in the hydrolysis product of HS-wool treated with 1,2-dibromoethane is thus explained.

Various Methods of Treatment

The formation of lanthionine from wool cystine by treatment with KCN has often been reported in the literature. It is, however, an innovation to find cysteic acid and another substance reacting with ninhydrin ($R_{lys} = 0.17$) in the hydrolysis product of wool treated with caustic soda. This indicates a difference in the reaction mechanisms of the decomposition of cystine by KCN and NaOH, respectively.

Conclusion

The results described herein of the systematic examination of wool samples by means of paper chromatography serve once again to demonstrate the significance of this method of investigation. Special attention should be given to two aspects of the method:

(*1*) Up to the present time, the paper chromatographic method provides the sole possibility of analyzing the various chemically modified wool samples. Since some of the methods have already passed into technical practice, the paper chromatographic analysis technique has now become an indispensible tool in testing wool samples.

(*2*) The results obtained with the paper chromatographic method have provided some valuable pointers for wool research from a protein standpoint. However, it will probably be some time before these results are turned to full account for preparative purposes. The most important finding is the surprisingly ready formation of lanthionine from wool, acetone, and bicarbonate. After the conclusion of the investigations described, Osterloh actually succeeded in preparing lanthionine in good yield from wool treated in this manner, so that the paper chromatographic findings have already been confirmed on a preparative scale in this particular case.

Summary

The chemically modified wool samples described in Part I of this series have been investigated by paper chromatography.

Deviations in behavior from the hydrolysis products of untreated wool were observed in the hydrolysis products of samples modified in the following manner: treatment with epichlorohydrin, acetylation, dinitrophenylation, nitration, formalization, treatment with Kaurit 140, butanediol-*bis*-chloromethyl ether, acetone and bicarbonate, *bis*-chloromethyldimethylbenzene, reduction and methylation, KCN and NaOH treatment.

In some cases the paper chromatographic method of investigation offers the sole means of analysis of chemically modified wool. Its significance for the chemistry of wool research has been demonstrated—*viz.*, the formation of lanthionine in wool treated with acetone and bicarbonate.

Acknowledgment

The author expresses his thanks to the following bodies for promoting wool research: Deutsche Forschungsgemeinschaft in Bad Godesberg, International Wool Secretariat in London and Düsseldorf, and Deutsches Wollforschungsinstitut e.V. at the Technical High School in Aix-la-Chapelle.

Thanks are due to Mr. L. Richards for translation of this paper.

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The Relative Responsiveness of Certain Properties of Cotton Fiber to Microbial Action

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Abstract

In continuation of an inquiry into the effect of microbial action and weathering on cotton fiber, experiments have been carried out to determine the relative responsiveness of certain fiber properties to the growth of fungi. Raw cotton fiber and unbleached cotton fabric were inoculated and incubated for varying periods up to 28 days in a water-vapor-saturated atmosphere with each of three common fungi under pure-culture conditions, and various properties of the fiber and fabric were measured subsequently. In general, the aqueous-extract pH and the reducing-substance content of the incubated samples changed quickly and substantially, the former increasing and the latter decreasing. Degree of swelling in 18% NaOH and Congo red values changed little during the first few days of incubation, but later increased progressively. Strength, as measured on fiber by the use of a Pressley breaker equipped with 1-mm.-gap jaws or on fabric by the 3-in. ravelled-strip method on a Scott tester, showed little or no decrease. Length, determined by the Suter-Webb sorter method, decreased moderately with one of the fungi, but little if at all with the other two. Wax content decreased, but only moderately. *Cladosporium*-incubated fiber became measurably and visibly darker. Absorption of the direct dye Chlorantine Fast Green by fiber incubated with *Cladosporium* and mercerized before dyeing increased markedly with increasing periods of incubation.

Incubation at 95% R.H. greatly slowed down the rate of change in pH and reducing-substance content as compared with results obtained in a saturated atmosphere and prevented change in the alkali-swelling response.

Introduction

In recent investigations on the deterioration of cotton, microbial action on the fiber has been shown to result in changes in aqueous-extract pH [5, 6, 11], content of water-soluble reducing substances [5, 6], and degree of swelling in alkali as measured by an alkali-centrifuge test [12]. In evaluating the results of tests for these properties, it is of interest in many cases to know the relative responsiveness of the three properties to microbial growth, not only in comparison with each other but also with respect to other important characteristics of the fiber.

Some investigators have been of the opinion that the strength of cotton is more highly responsive to microbial action than are other properties of the fiber. Blum and Stahl [2], for example, stated: "It has long been known that when fungi degrade cotton, the tensile strength decreases at a rapid rate. Other properties (except a few noted under the microscope), however, change little or not at all." A limited degree of support for this belief may be found in certain earlier papers. Rogers, Wheeler, and Humfeld [14] showed the high degree of responsiveness of strength loss in comparison with weight loss of incubated cotton fabric, while various authors have found the conventional cellulose viscosity methods to record little or no change as a re-

sult of microbial action sufficient to reduce the strength greatly ([15], pp. 295–7). Data presented herein indicate, however, that several properties of cotton fiber are, under the conditions described, distinctly more responsive to microbial action than is strength. It has also been found that fiber length losses, as determined by the Suter-Webb method, may be relatively small and difficult or impossible to measure under conditions in which other properties show major and easily measurable changes. Aqueous-extract pH and copper-reduction values are both noteworthy for their extremely high responsiveness to microbial action.

In the present investigation, cotton samples were incubated with microorganisms under controlled pure-culture conditions in the laboratory, and certain of their properties were measured subsequently. By studying the effect of microbial action on fiber properties under such relatively simple circumstances, it was thought that one might obtain information applicable to the analysis of fiber deterioration problems in the more complex situations which often prevail under practical conditions. Progress along this line of endeavor has already been made in the application of the aqueous-extract pH test to fiber weathered in the field prior to harvest [11]. In view of the well-established fact that microbial physiology frequently varies considerably from one organism to the next, three different species were used in the present work in attempting to arrive at reliable conclusions. All are known to be of common occurrence on cotton in the field [9, 10], while at least one—the *Fusarium*—also occurs frequently on cotton in humid storage [9].

Methods

The following description of methods is condensed for the sake of brevity. A mimeographed supplement providing a more detailed description is available from the authors upon request.

Incubation Procedures

The incubation procedure was the same as that used in a previous study [11], with the minor difference that the raw fiber was supported in the incubation bottles with glass rods instead of glass beads. In this procedure, sterilized raw fiber or unbleached fabric strips are dusted with dry spores, and incubated in pure culture in culture bottles over water or over a mineral-salts solution to control the relative humidity. The isolates used were *Cladosporium* #113, *Fusarium moniliforme* #103, and *Alternaria* #122.

Aqueous-Extract pH

As in previous work [11], this determination was performed by kneading with a glass rod for about 30 sec. either 0.5 g. or 1.0 g. of fiber or fabric with 7.5 cc. or 15.0 cc. of freshly distilled water, respectively, and determining the pH of the liquid so obtained on a Beckman glass-electrode pH meter.

Copper-Reduction Values

The method used was essentially that of Heyes [7], the only major deviation from his procedure being that the heating of the fiber with the alkaline copper-containing solution was carried out at a lower temperature and for a shorter time period—namely, 85°C for 1 hr.

This test was performed on both unextracted and water-extracted samples of 250 mg. weight. In the latter case, the analysis was run on samples which had previously been weighed to 250 mg. and then extracted with water in micro-Soxhlet extractors for 3 hrs. The values obtained on unextracted samples are referred to as "total copper-reduction values," those on extracted samples as "extracted-sample copper-reduction values," and the difference between these two as "water-soluble copper-reduction values."

Kendall Copper Index (ESRM Test)

This test was carried out as described by Hall and Elting [6]. It is a copper-reduction test which involves the addition to a hot-water centrifugate from a fiber sample of a proprietary urinanalysis (Clinitest) tablet, and the visual estimation of reducing-substance content from the shade of the precipitate formed.

Picric Acid Values

The reagents used were as follows: (*1*) Solution *A*—Add 18 g. picric acid to 250 ml. of 1% NaOH in a 500-ml. volumetric flask. Add 200 ml. of hot water. Cool to room temperature and add water to the mark. (*2*) Solution *B*—Place 25 g. of Na_2CO_3 in a 100-ml. volumetric flask and add water to the mark.

Ten milliliters of distilled water, brought to a temperature of 85°C, was poured over 250 mg. of cotton in a Conrad micro-copper-number tube. The tube was placed in a constant-temperature bath at 85°C for 1 hr., with stirring of the cotton every 15 min. After the extract had cooled to room temperature, 5 ml. of it was pipetted into another micro-copper-number tube, and 5 ml. of solution *A* and 1 ml. of solution *B* were added. The tubes were placed in a constant-temperature bath at 85°C, and color development was allowed to take place for 23 min. (it took 3 min. for the solution to reach the temperature of the bath). The tubes were then removed from the bath and cooled to room temperature, and the depth of color was measured in an Aminco colorimeter, Type F, using 50-ml. tubes and a # 53 (yellowish green) filter. The colorimeter reading was converted to "equivalent percent glucose" or "picric acid value" by reference to a calibration curv. obtained with pure glucose.

Congo Red Values

This test was conducted by a method similar to that described by Bright [3]. The fibers were examined for the presence of red spiral bands, presence of localized red areas, and presence of even red staining throughout half or more of the length of the fiber examined. The total percentage of fibers falling into these three classes was designated as the "total Congo red value." Normal, undamaged fiber contains a very low percentage of fibers which stain distinctly red throughout, and these are found to be thin-walled, convoluted fibers which are easily recognizable. Disregarding these fibers, the remaining deep-red-staining fibers were counted—*i.e.*, fibers staining deep red for more than half of the area visible in the field. The percentage of these fibers was designated as "percent red-stained fibers."

Figure 1 illustrates the role of the primary wall in the Congo red test.

Alkali-Swollen Fiber Width

Samples were swollen by immersion in 18% NaOH at 20°C for 15 min., mounted in 18% NaOH on a microscope slide, and measured by the use of a compound microscope equipped with a 7.5 × movable-scale micrometer eyepiece and a 4-mm. objective. Each fiber was measured at the point of maximum width visible within the field. The figures in the tables are averages of measurements on 250 fibers.

Alkali-Centrifuge Values

This test was carried out according to a standardized method previously described [12].

Reflectivity of Visible Light

About 100 mg. of fiber was placed, without combing, into the reflectivity cell of a Beckman DU spectrophotometer under a 24 mm. × 50 mm. No. 1 "Crystal" microscope cover glass, and reflectivity readings were taken at 5,000 and 7,000 Ångström units in the usual manner.

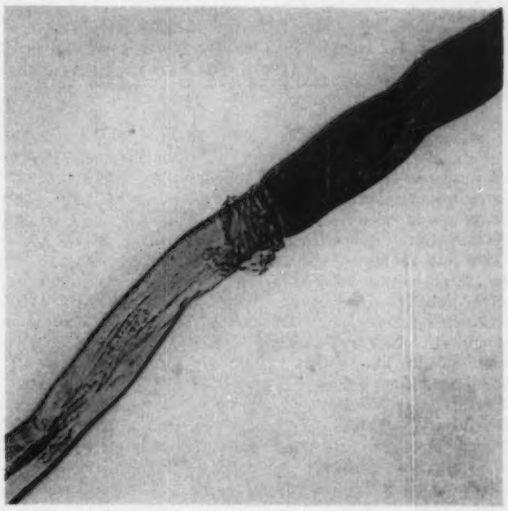

FIG. 1. *Photograph demonstrating the fact that the primary wall of the cotton fiber is very little stained or penetrated by the dye Congo red, whereas the secondary wall stains readily. By gentle scraping with dull forceps, the primary wall of an undried fiber has been caused to slip along the outside of the fiber to a point near the center of the section illustrated, where it may be seen in the form of a wrinkled collar. The fiber was then subjected to the Congo red test. Note the deep dyeing of the secondary wall where the primary wall has been removed, and the failure of the dye to penetrate through the primary wall in the remainder of the fiber section shown. Failure of dye penetration through the primary wall is not due to waxy constituents of the same, since thorough dewaxing, as for wax analysis, causes no increase in the dye penetration. (Photo by M. Jaeger.)*

Pressley Strength

Fiber strength determinations were made in the conventional manner on a Pressley strength tester ([1], p. 268) at 70°F and 65% R.H., with the exception that jaws ground out at the interface to allow a 1-mm. breaking span were used. Each figure in the tables is an average of 10 replicate determinations.

Fiber Length (Suter-Webb)

This test was carried out in the usual manner at 70°F and 65% R.H. ([1], p. 252), care being taken that no fibers were discarded during the operation.

Percent Wax

These determinations were carried out according to the method of Conrad [4], with slight variations as previously described [8].

Fabric Breaking Strength

This test was made at 70°F and 65% R.H. in the customary fashion on a pendulum-type Scott tester with a jaw gap of 3 in. Leather facings were used on the jaws. The samples were 5½ in. in length and had been ravelled to 1 in. in width prior to incubation. Each figure in the tables is an average of 15 replicate determinations.

Experimental Results

The first experiments were concerned with incubation of the fungus *Cladosporium* sp. on fiber and fabric in an atmosphere saturated with water vapor (Table I). From the data on fiber, attention is called to the following points in the table. The aqueous-extract pH rose rapidly to a maximum in the range of pH 10; this property was obviously

TABLE I. EFFECT OF *Cladosporium* SP. ON VARIOUS PROPERTIES OF COTTON FIBER AND FABRIC INCUBATED IN A WATER-VAPOR-SATURATED ATMOSPHERE WITH THE FUNGUS IN PURE CULTURE

Type of test	Test result after following number of days incubation: 0	2	3	4	7	9	11	14	21	28
					Fiber					
pH (aqueous-extract)	6.3	7.6	8.5	9.5	10.0	10.2	10.1	10.1	10.1	10.1
Cu-reduction, total*	.72	.69	.54	.51	.33	.28	.20	.18	.16	.15
Cu-reduction, water-soluble	.69	.64	.50	.48	.29	.24	.15	.13	.13	.11
Kendall copper index (ESRM Test)	8	7	6	5	4	3	2	1	1–	1–
Picric acid values	.44	.42	.40	.37	.34	.32	.30	.28	.28	.25
Congo red values, total										
Uncombed†	24	17	24	29	57	75	80	90	93	93
Combed	24	—	—	—	51	—	—	90	93	90
Congo red values, red-stained‡	0	1	1	5	25	39	52	69	80	83
Alkali-swollen fiber width	27.5	—	—	—	29.4	—	—	32.1	35.7	36.8
Alkali-centrifuge values**	185	191	192	198	288	244	282	317	317	323
Reflectivity, 5,000 Å.	65.5	66.3	66.2	66.9	60.4	57.1§	54.3	51.3	49.7	51.8
Reflectivity, 7,000 Å.	92.7	91.7	91.8	89.8	86.6	79.3	78.5	73.7	71.6	72.9
Pressley strength	4.98	—	—	—	5.01	—	—	5.05	5.17	5.03
Mean fiber length, in.	.92	—	—	—	.87	—	—	.89	.87	.88
(Suter-Webb, duplicate tests)	.91	—	—	—	.91	—	—	.89	.88	.84
Percent wax	0.68	—	—	0.67	0.63	—	—	0.61	0.62	0.63
					Fabric					
pH (aqueous-extract)										
Inoculated and incubated	7.4	10.0	10.1	10.1	10.2	10.1	10.1	10.1	10.1	10.1
Incubated sterile	7.4	7.5	7.5	7.4	7.5	7.4	7.4	7.3	7.2	7.2
Cu-reduction, total, inoc. and incub.	.22	.09	.08	.07	.07	.07	.06	.07	.05	.08
Cu-reduction, water-soluble, inoc. and incub.	.20	.07	.05	.04	.04	.04	.02	.04	.02	.04
Alkali-centrifuge values	223	223	243	255	294	307	308	311	310	302
Breaking strength (Scott 3 in.)										
Inoculated and incubated	—	108	108	108	110	108	110	108	111	113
Incubated sterile	106	109	107	108	109	108	109	107	109	114

* Average deviation from mean of duplicate values whose average recorded here was .0063.

† Control samples incubated under sterile conditions for periods of 0, 1, 2, 3, and 4 weeks yielded values of 18%, 20%, 20%, 20%, and 22%, respectively.

‡ A check on these values run independently by a different operator yielded percentages deviating by 0, 0, 2, 0, 0, 9, 7, 3, and 5 from the values obtained by the first operator for incubation periods of 2–28 days, respectively.

** Sterile control samples incubated for periods of 0, 1, 2, 3, and 4 weeks yielded values of 185, 182, 186, 185, and 185, respectively.

§ Darkening in color was evident to the eye on samples incubated for 9 days or longer.

highly responsive to the growth of the fungus. Total copper-reduction values decreased rapidly, as did also water-soluble copper-reduction values, the latter being regularly but only slightly lower than the former. The Kendall copper index decreased. Picric acid values also decreased regularly, but not as rapidly as the copper indices. Congo red values, alkali-swollen fiber width, and alkali-centrifuge values all increased, while reflectivity decreased progressively.

In contrast to the properties just mentioned, the Pressley strength changed very little. It was thought at first that the validity of the Pressley strength results might be somewhat doubtful, for the reason that weakened fibers might have been selectively combed out during preparation of the incubated samples for test. However, when samples were combed in the same manner and then both combed and uncombed samples subjected to the Congo red test, it was found that the Congo red values obtained on the combed samples were not materially different from those on the uncombed samples (Table I), thus indicating that the above-mentioned possible objection to the Pressley strength results is in all probability not valid. Percent wax showed relatively minor decreases.

In the tests on fabric strips incubated with *Cladosporium* (Table I), the aqueous-extract pH rose very rapidly, copper-reduction values decreased, and alkali-centrifuge values increased, while breaking strength showed little if any significant change with increasing incubation, and sterile-incubated control samples exhibited almost exactly the same strength as the inoculated and incubated samples. For unknown reasons, the initial copper-reduction value on the fabric was low as compared with the corresponding value for fiber. The cotton in the fabric may have been subjected to microbial action at some previous time, although there are also other possible explanations (see p. 38).

TABLE II. EFFECT OF *Fusarium moniliforme* ON VARIOUS PROPERTIES OF COTTON FIBER AND FABRIC INCUBATED IN A WATER-VAPOR-SATURATED ATMOSPHERE WITH THE FUNGUS IN PURE CULTURE

Type of test	Test result after following number of days incubation: 0	2	3	4	7	9	11	14	21	28
					Fiber					
pH (aqueous-extract)	6.3	8.5	9.7	10.1	10.2	10.3	10.2	10.2	10.2	10.2
Cu-reduction, total*	.72	.47	.37	.15	.13	.12	.10	.11	.06	.05
Cu-reduction, water-soluble	.69	.44	.33	.13	.10	.07	.06	.08	.03	.02
Kendall copper index (ESRM Test)	8	7	5	4	3	3	2	1	1−	1−
Picric acid values	.45	.39	.31	.21	.19	.18	.18	.18	.18	.15
Congo red values, total										
Uncombed†	24	20	24	26	80	84	88	90	92	95
Combed	24	—	—	—	90	—	—	94	95	93
Congo red values, red-stained	0	0	1	5	59	69	56	75	77	66
Alkali-centrifuge values	185	191	195	208	291	310	287	313	313	302
Pressley strength	4.81	—	—	—	4.81	—	—	5.07	4.81	5.11
Mean fiber length, in.	.90	—	—	—	.87	—	—	.87	.89	.88
(Suter-Webb, duplicate tests)	.91	—	—	—	.90	—	—	.89	.90	.88
Percent wax	0.68	—	—	0.64	0.63	—	—	0.61	0.61	0.64
					Fabric					
pH (aqueous-extract)										
Inoculated and incubated	7.4	10.1	10.2	10.2	10.1	10.1	10.0	10.2	10.0	10.2
Incubated sterile	7.4	7.4	7.4	7.4	7.3	7.2	7.3	7.4	7.3	7.3
Cu-reduction, total, inoc. and incub.‡	.22	.09	.08	.08	.07	.06	.06	.05	.03	.04
Cu-reduction, water-soluble, inoc. and incub.	.20	.05	.05	.05	.03	.04	.03	.03	.00	.01
Alkali-centrifuge values	223	240	244	269	249	263	260	296	286	285
Breaking strength (Scott 3 in.)										
Inoculated and incubated	—	106	104	103	107	107	106	108	110	111
Incubated sterile	106	103	105	104	106	107	107	107	111	109

* Average deviation from mean of duplicate values whose average recorded here was .0064.

† A check on these values run independently by a different operator yielded percentages deviating by −1, 3, 4, 0, 0, −8, −4, −1, and −3 from the values obtained by the first operator for incubation periods of 2–28 days, respectively. For the red-stained fiber values the independent checks deviated by 1, 0, 0, −5, 5, −1, −1, 1, and 2 from the values obtained by the first operator.

‡ Total Cu-reduction values for samples incubated sterile for all time periods were all in the range of .21–.24.

Results of an experiment in which *Fusarium moniliforme* was incubated on cotton fiber and fabric are shown in Table II. In the tests on the fiber, the aqueous-extract pH increased very rapidly. Copper-reduction values and the Kendall copper index decreased, as did also the picric acid values, while the Congo red values and the alkali-centrifuge values increased. All of these properties were more responsive to fungus growth than either the Pressley strength or fiber length, neither of which showed clearly significant changes. Again, combed and uncombed samples yielded essentially similar results in the Congo red test. Wax content decreased slightly.

The results on fabric incubated with *Fusarium* were similar to the *Cladosporium* results on fabric in the sense of exhibiting large changes in pH, copper-reduction values, and alkali-centrifuge values, with no accompanying loss in strength and no consistent difference in strength between inoculated samples and sterile-incubated controls. The *Fusarium* differed from the *Cladosporium* in that, unlike the latter, it produced very little visible change in color of the fiber or fabric.

Results of tests on fiber and fabric incubated with *Alternaria* sp. are to be seen in Table III. With this fungus, although the aqueous-extract pH and the copper-reduction indices of the fiber changed rapidly, the Congo red values and alkali-centrifuge values both changed somewhat less rapidly than with the *Cladosporium* and *Fusarium*. Pressley results again showed no indication of loss in strength, while mean fiber length was essentially constant throughout the course of the incubation. Wax content decreased slightly. In fabric tests, marked changes in aqueous-extract pH and in copper-reduction values were again unaccompanied by changes in strength, and again no difference in strength could be detected between inoculated and incubated samples and sterile-incubated controls, in spite of large differences between the two sets in aqueous-extract pH.

Although the alkali-centrifuge test has been shown to be definitely responsive to fungus growth under the conditions of Tables I, II, and III, it is equally apparent upon examination of the same tables that increases in this property lagged behind the responses shown in the pH and copper-reduction tests. This difference in responsiveness between the pH and copper-reduction tests on the one hand and the alkali-centrifuge test on the other was accentuated in another experiment (Table IV) in which the incubations were carried out at 95% R.H. instead of in a saturated atmosphere. Inoculation and other features of the test were the same as for the tests of Tables I, II, and III, with the exception of the decreased humidity and the fact that all tests were run at 20°C. It may be seen that under the conditions

TABLE III. EFFECT OF *Alternaria* SP. ON VARIOUS PROPERTIES OF COTTON FIBER AND FABRIC INCUBATED IN A WATER-VAPOR-SATURATED ATMOSPHERE WITH THE FUNGUS IN PURE CULTURE

Type of test	Test result after following number of days incubation: 0	2	3	4	7	9	11	14	21	28
					Fiber					
pH (aqueous-extract)	6.3	7.6	9.3	9.8	10.2	10.2	10.2	10.2	10.2	10.2
Cu-reduction, total	.72	.58	.44	.33	.13	.12	.10	.15	.09	.10
Cu-reduction, water-soluble	.69	.54	.41	.29	.10	.09	.07	.11	.06	.08
Kendall copper index (ESRM Test)	8	6	5	3	2	1	1	1−	1−	1−
Picric acid values	.46	.40	.34	.32	.26	.20	.20	.20	.20	.20
Congo red values, total	24	18	23	26	35	38	41	42	51	62
Alkali-centrifuge values	185	188	190	192	196	206	206	212	219	217
Pressley strength	5.08	—	—	—	5.04	—	—	5.33	5.25	5.17
Mean fiber length, in.	.90	—	—	—	.91	—	—	.91	.91	.90
(Suter-Webb, duplicate tests)	.90	—	—	—	.91	—	—	.90	.92	.91
Percent wax	0.68	—	—	0.65	0.63	—	—	0.62	0.61	0.61
					Fabric					
pH (aqueous-extract)										
Inoculated and incubated	7.4	9.9	10.0	10.0	10.0	9.9	10.0	9.9	10.0	10.0
Incubated sterile	7.4	7.4	7.4	7.4	7.3	7.2	7.3	7.4	7.3	7.3
Cu-reduction, total, inoc. and incub.	.22	.09	.08	.07	.05	.05	.05	.04	.04	.06
Cu-reduction, water-soluble, inoc. and incub.	.20	.06	.05	.04	.02	.02	.02	.02	.02	.03
Breaking strength (Scott 3 in.)										
Inoculated and incubated	—	109	107	108	109	109	111	111	110	110
Incubated sterile	107	103	105	104	106	107	107	107	111	109

of the experiment of Table IV, not only did a very considerable decrease in the rate of change of the pH and copper-reduction values occur, but also the alkali-centrifuge test showed no response at all. The lack of response in the alkali-centrifuge test within the period of the experiment is presumably a result of the greatly decreased rate of growth of the fungi under these conditions and the fact that the fungi grow first and preferentially on the noncellulosic fiber constituents and during this early growth-phase produce little or no *S*-factor [13]. It is also recognized as a possibility that decreases in humidity cause the fungi to grow more exclusively in the lumen, in which location they would presumably exert little effect on the alkali-centrifuge response, which is thought to be principally associated with the outer wall of the fiber [12].

TABLE IV. EFFECT OF INCUBATION AT 95% R.H. IN THE PRESENCE OF THREE FUNGI IN PURE CULTURE ON AQUEOUS-EXTRACT pH, COPPER-REDUCTION VALUES, AND ALKALI-CENTRIFUGE VALUES OF RAW COTTON *

Micro-organism	Weeks of incubation	Aqueous-extract pH	Copper-reduction values (total)	Alkali-centrifuge values
Cladosporium #113	1	6.9	.50	207
	2	8.6	.40	209
	3	8.9	.40	211
	4	9.9	.20	216
Fusarium #103	1	6.7	.57	209
	2	10.1	.17	214
	3	9.1	.35	212
	4	9.6	.32	206
Alternaria #122	1	6.5	.55	206
	2	7.7	.38	205
	3	9.0	.35	206
	4	10.2	.09	207

* Note absence of change in alkali-centrifuge values and much decreased rate of change in the other two properties in comparison with the results shown in Tables I, II, and III.

TABLE V. STRENGTH AND AQUEOUS-EXTRACT pH OF UNBLEACHED COTTON DUCK DURING STORAGE AFTER PREVIOUS INCUBATION WITH *Aspergillus oryzae* IN A WATER-VAPOR-SATURATED ATMOSPHERE FOR 7 DAYS AND ALSO OF UNINCUBATED CONTROL STRIPS

Period of storage	Aqueous-extract pH		Breaking strength (lbs.)	
	Incubated and stored	Control stored	Incubated and stored	Control stored
no storage	9.3	7.2	104	108
1 mo.	8.9	7.2	108	108
3 mos.	9.1	7.4	108	106
6 mos.	8.6	7.2	105	108
9 mos.	8.5	7.4	108	104
12 mos.	7.7	6.9	104	104

The effect of fungus incubation on the fiber's absorption of dye was measured not only by microscopic fiber counts and the dye Congo red, but also by colorimetric determinations of dye loss from a solution of Chlorantine Fast Green in contact with the mercerized fiber. In this experiment (Figure 2) unincubated fiber and fiber incubated for varying time periods in a saturated atmosphere with *Cladosporium* were swollen in 18% NaOH solution, rinsed in tap water, neutralized with 3% acetic acid, rinsed in tap and distilled water, and then blotted dry. Samples of an original 500-mg. weight were placed into 25 cc. of a solution containing .02% dye and .05% Na_2SO_4 at 80°C. One-cc. samples were removed from the bath after 15 min. of dyeing and diluted to 10 cc., and the dye concentration was measured photometrically in a Lumitron. The results, in terms of percent dye on the fiber, are shown in Figure 2.

Although strength was seen from the experiments of Tables I, II, and III to be remarkably unresponsive to fungus growth by comparison with several other properties, it was considered as a possibility

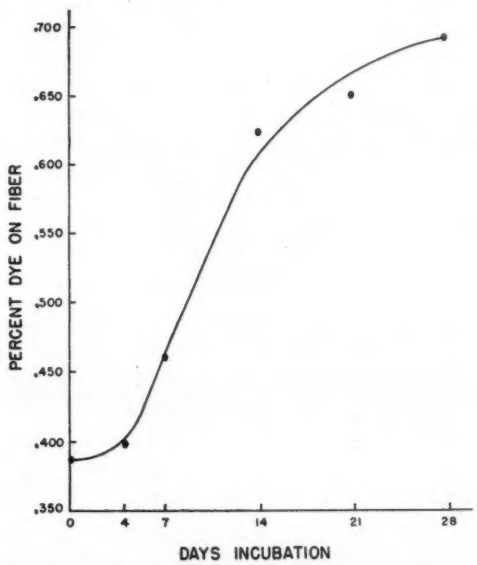

FIG. 2. *Effect of incubation of cotton in a saturated atmosphere with* Cladosporium *on its absorption of Chlorantine Fast Green. The fiber was mercerized before dyeing.*

that the existence of a high pH condition might possibly lead to losses in strength during subsequent storage—*i.e.*, that there might be a delayed effect of the microorganism on strength. To test this possibility, a series of unbleached fabric strips was raised to a high pH by fungus incubation, stored at room temperature and humidity, and tested at intervals for both strength and aqueous-extract pH. The results (Table V) showed no delayed effect on the fabric strength during storage.

Discussion

Although it is clearly apparent from the data of Tables I–IV that fungi can bring about a decrease in the reducing-substance content of cotton fiber, the writers wish specifically to avoid implying that a low reducing-substance content in raw fiber necessarily is a result of prior microbial action. Hall and Elting [6] pointed out that the reducing-material content varies widely for different cottons and that one of the reasons for low content can be leaching by heavy dews or rainfall. A low content of reducing sugar in the fiber could also be ascribed to metabolic activities of the living fiber itself in the period of its later development and senescence—polymerization of the sugar into cellulose, other types of transformation into nonreducing compounds, and respiration. It is advisable to consider these different possibilities in the interpretation of the Kendall copper index (ESRM test) on raw cotton samples of unknown origin. Work on this and other phenomena described in the report of Hall and Elting [6] is currently under way in an attempt to elucidate some of the problems of microbial action on raw cotton as presented by these authors.

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The Relation Between External Shape and Internal Structure of Wool Fibers

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Abstract

Relations between the twisted bilateral structure of the cortex of the wool fiber and its external shape are pointed out. Both the crimped form and the elliptical cross-sectional shape follow the twisted internal structure. Changes in crimp shape are interpreted as a consequence of the unequal expansion of the ortho- and the paracortex. The geometrical relations of the crimp and ortho- and paracortex are traced back to events occurring during keratinization of the fiber.

RECENTLY evidence has been given to show that the cortex of wool fibers may be divided into two approximately equal halves differing in stability and reactivity [4, 5]. The less stable, more reactive half has been referred to as the "orthocortex," and the other, the "paracortex." † Further work has done much to confirm this hypothesis, and it is clear that this concept of fiber structure promises to cast light on many of the peculiarities of wool. In the present article the relation between the internal structure of the cortex and certain external geometrical aspects of the fiber will be discussed briefly.

The Crimped Form and Cross-Sectional Shape

Horio and Kondo [4] and the present writer [5] have described a connection between the crimped form and the structure of the cortex. Briefly, the two hemicylinders of ortho- and paracortex are twisted together from one end of the fiber to the other, and the pitch of the twisted structure is equal to the wave length of the crimp (Figure 1). Whether the direction of twist remains the same or alternates is difficult to decide, but the reactive orthocortex is always found on the convex side of the cortical filament—*i.e.*, it is on the outside face at the crests and troughs of the waved form—and between each crest and trough it crosses from one side of the fiber to the other.

Wool fibers are rarely circular in cross section and many, to a first approximation, may be regarded as elliptical. On moving along a fiber, the axes of the elliptical section are found to oscillate. Many years ago Rossouw [8] demonstrated a relation between the progression of the axes and the waved form, which immediately suggests that there is a connection between the twisted cortical structure and the shape of cross sections. According to Rossouw, the rotation of the axes has the same period and is in phase with the waved form. At crests and troughs the major axis is at right angles to the plane of the waved form (regarded as approximately in one plane). It would thus seem that the major axis of

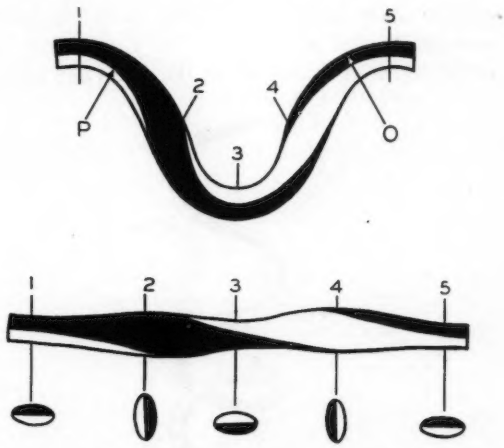

FIG. 1. *The relation between crimp form, cross-sectional shape, and the internal structure of the cortex;* O = *orthocortex (less stable and more reactive), and* P = *paracortex. The straightened form (center) shows more clearly the relation between the axes of the elliptical cross section and the twisted cortex.*

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† "Ortho" in the sense of "regular" or "normal," and "para" meaning "along with" or "at the side of."

the cross section lies in the surface separating the ortho- and the paracortex and that the rotation of the external form is determined by the internal structure. Confirmation of this deduction was obtained by examining the cross sections of fibers dyed to define the orthocortex [7]. In about 80% of the definitely noncircular sections the orthocortex and paracortex were found to lie on either side of the major axis. Figure 1 has been constructed to show these relations in the idealized case. A similar relation has been found in a sample of negro hair, although in this case the location of the orthocortex had to be determined by a stability test [5] since staining was ineffective.

The Crimped Form and Other Shapes

The regular crimped form approximates a sine curve, but is more complex and three-dimensional [8, 9]. However, the various forms assumed by a fiber may be related readily, and changes in these forms understood more easily, when the internal structure is considered.

First, we shall describe the geometrical derivation of several familiar forms from a simple spiral, and, subsequently, consider whether these ideal relations have any physical implication.

Consider a spiral (Figure 2) in which the orthocortex is everywhere on the convex face of the filament. A simple waved form is obtained from this, as Horio and Kondo have pointed out, by a deformation which involves a twisting of the filament at such points as *A*,*A'*, etc. The orthocortex thus comes to lie on the outside at the crests and troughs, and a twist appears between each crest and trough, as is actually found. On assuming an originally elliptical filament, the relation of the cross-sectional shape and crimp is correctly reproduced. Horio and Kondo have discussed the geometry of such transformations in much detail, and reference may be made to their article [4].

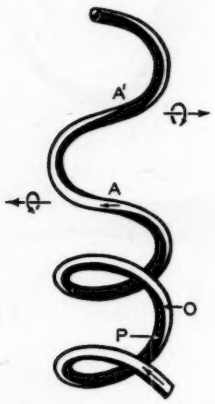


FIG. 2. *A possible mechanism for the formation of a wave form by deformation of a spiral. (See also Horio and Kondo* [4].)

Various distorted waved forms, probably nearer to the forms actually assumed by the fibers, may be derived by considering the changes in curvature which follow a relative extension or contraction of the elements of a bilateral filament (Figure 3). A simple preferential increase in length of the orthocortex increases the curvature of short elements of lengths of the filament (Figure 3, *A* and *B*). Starting with a simple planar sine curve (Figure 4), the crests (*B*) and troughs (*C*) become more pinched, and at the twisted zones (*A*,*A'*,*A''*, etc.) the filament alternately bends into the paper (*A*,*A''*, etc.) and out of the paper (*A'*,*A'''*, etc.). The waved form so produced (Figure 7*A*) approaches that normally assumed by a fiber [8]. On reversing the length changes, the original waved form is recovered.

Woods [9] described another, more complex waved form, produced by relaxing the fiber to remove internal strains, which he regards as the "struc-

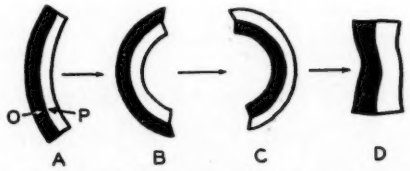


FIG. 3. *Successive changes in curvature of a short element of the bilateral cortex produced by a relative expansion (or contraction) of the orthocortex:* A → B, *longitudinal expansion of orthocortex (very small);* B → C, *supercontraction of orthocortex (curvature is reversed);* C → D, *swelling of orthocortex and/or supercontraction of paracortex (fiber is straightened).*

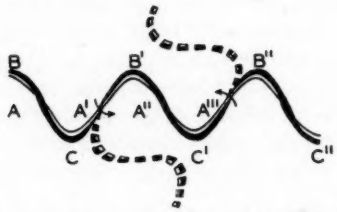


FIG. 4. *The formation of Woods' structural form* [9] *by the removal of alternate twists* A',A''', *etc., from the twisted cortex. (See also the photograph of the model, Figure* 7B.)

tural" or primordial form originally acquired in the follicle. This shape can be regarded as a partial conversion to a spiral form in which every second twist of the series (*A*,*A*", etc., in Figure 4) is removed. Photographs of a model (Figure 7*B*) show this more clearly. Further untwisting produces a complete spiral, which may thus seem to be even more "relaxed" than Woods' "structural" form. Some of Freney's coils [2] may have been of this type.

Many of the twistings and writhings of wool fibers could be of this nature. Such movements may be considered as normal, since they occur in conditions of changing humidity, laundering, felting, etc., and do not lead to permanent damage. It will be noted that in all these forms the orthocortex always lies on the convex face of the curved filament (Figure 3*B*) and is thus always longer than the paracortex. The physical implications of these relations will be discussed below.

Coils and Spirals

Further distorted forms, described by Freney [2], result when the orthocortex preferentially supercontracts (Figure 3, *B* and *C*) to a much shorter length than the paracortex. In such cases the curvature is reversed, the shorter orthocortex now occupying the concave face of the filament.

When the fiber is held slack at the ends, coils result [2]; when it is confined to a plane—*e.g.*, between cover glass and microscope slide—flat spirals form (Krais-Viertel Test, Figure 5). Should the paracortex subsequently contract or the orthocortex swell, the filament may straighten out (Figure 3*D*) and the coils or spirals open out.

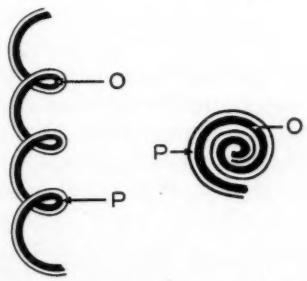


FIG. 5. *Coils and spiral resulting from the supercontraction of the orthocortex, which now forms the concave face of the filament.*

Twisting

The twisting of a fiber held at one end and stretched taut by means of a weight or the localized internal twisting noted in fibers held taut at each end should be relatable to adjustments between the lengths of ortho- and paracortex. However, little information about these phenomena is available. The twisting of the unloaded free end of a suspended fiber has been studied very thoroughly by Goldsworthy and Lang [3], but, unfortunately, this type of movement is not easily interpreted. In a perfectly symmetrically waved form a relative change in the lengths of ortho- and paracortex would lead to the shape changes described above, and there would be a more or less slight movement of the free end depending on where the cut occurs in the final wave. The substantial twisting noted in practice seems to be a result of the accumulated effect of accidental irregularities in the waved form (or of the underlying internal structure), but one cannot even assert that the more irregular the fiber the greater the twist, since the final twist is the resultant of many positive and negative contributions. It seems that only a step-by-step examination of the changes in shape along the length of the fiber can give an account of the observed twist in any special case.

The Original Shape of the Fiber

In the above discussion, the derivation of the shapes of model fibers from a spiral was considered in a purely ideal, geometrical sense. However, the simplicity of these relations suggests the possibility that the spiral form is the original form of the fiber as it emerges from the follicle and that the waved

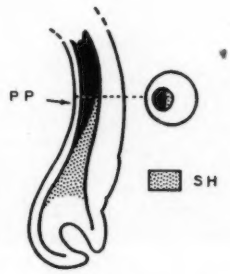
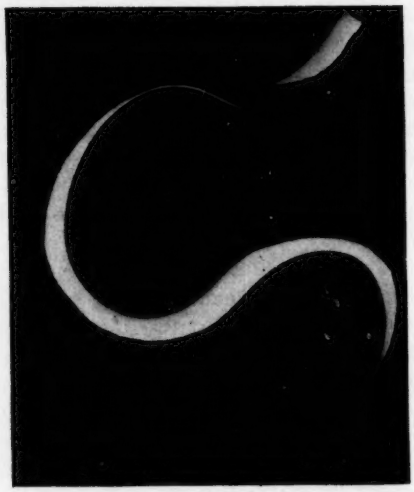
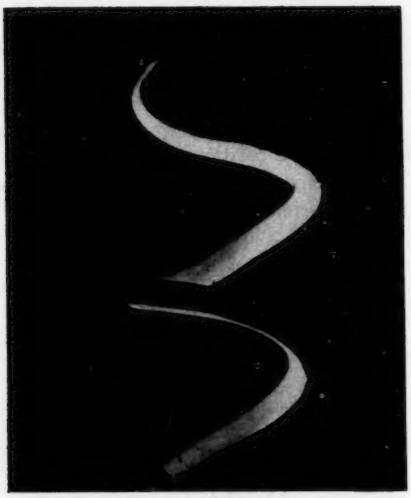


FIG. 6. *Suggested relation between the shape of the follicle in the keratinization zone and the formation of the ortho- and paracortical differentiation by an unsymmetrical keratinization. Keratinization begins and is assumed to be more complete on the concave side of the follicle (presumptive paracortex,* PP).

A



B



C

FIG. 7. *Photographs of models of fiber forms. (Black, orthocortex; white, paracortex.)* A—*Three-dimensional waved form developed from a sine curve by the small preferred lengthening of the orthocortex. This curve is similar to the actual shape assumed by wool fibers in the fleece;* B—*Woods' "structural" form;* C—*Spiral developed by removing further twists from* B.

form develops subsequently, as suggested by Figure 2; this view has been argued by Horio and Kondo [4]. Earlier, Woods [9] proposed the shape in Figures 4 and *7B* as the original form, since the fiber seemed to revert to this shape when internal strains were removed. It seems possible that this form is only "half" relaxed—*i.e.*, half of the twists *A*,*A*", etc., is removed—and that the fully relaxed form is the spiral. Horio and Kondo [4], from an analogy to crimped rayon, favor the spiral form, and they discussed the mechanism producing a transformation of the type shown in Figure 2. They analyzed in some detail the dynamic aspects of the problem—i.e., the interfiber forces within the fleece which, operating on the individual fibers, could cause them to assume the quasi-regular waved form observed in a staple. Reference may be made to their paper for a discussion of these matters.

From the point of view of the formation of the fiber in the follicle, the spiral form is plausible. Wool fiber follicles are, in fact, curved, and it seems clear that any short length of fiber must originally have had the curved shape of that portion of the follicle—the keratinization zone—in which it set (Figure 6). Whether the curvature is constant, necessitating the emergence of a spiral, or whether an alteration of curvature occurs, giving rise directly to a waved form, are questions which cannot be answered at present, but which are fundamental to an understanding of the formation of crimp. If an oscillation of the follicle [1] occurs, there would be no need to look for a transformation of the fiber into a waved form after emergence, and the interfiber forces discussed by Horio and Kondo would be confined to establishing some degree of uniformity and regularity in the existing waves.

Auber [1] has shown that in the follicle of the wool fiber keratinization is unsymmetrical, and elsewhere [6] reasons will be given, based on the difference in cystine content and staining reactions of the two components, to suggest that the ortho-para differentiation is due to uneven keratinization. Hardening commences on the side of the follicle where the outer root sheath is thinnest, and this side is also the *concave* side of the fiber. When it is further assumed that keratinization is more fully developed on the concave face, where it commences, this face becomes the paracortex, and the convex face becomes the orthocortex, which is the relationship observed in the emerged fiber.

Acknowledgment

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Molecular Weight of α-Cellulose from Jute and Allied Long Fibers

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Abstract

Four methods commonly employed for the estimation of cellulose, with special reference to jute, have been critically studied by means of viscosity measurements (and hence molecular weight) of the α-cellulose obtained with each in cuprammonium hydroxide. The cellulose molecule is degraded to different extents by all the methods of delignification; the Cross and Bevan method, however, appears to give the least degraded product. Pure cellulose is much more degraded than lignocelluloses. Generally, the use of caustic soda higher in concentration than 9.3% to extract α-cellulose or mechanical action of disintegration of the material brings about further degradation. The molecular weights of α-cellulose from different fibers have been determined. From the results thus obtained, a formula is suggested to calculate the intrinsic viscosity, and hence molecular weight, from the relative viscosity of a cellulose solution at a single concentration.

Introduction

The polysaccharides of long vegetable fibers consist mainly of α-cellulose and hexosans, pentosans, and polyuronic acids, comprising hemicelluloses, which are short-chain molecules. Cellulose is not homogeneous, being an aggregate of molecules of different chain lengths. Molecular weight or degree of polymerization (D.P.) of a product determined from its viscosity, therefore, represents an average value. D.P. of α-cellulose may vary *in vivo* at different stages of development of the plant [15] and also with sources.

It is still an open question whether or not xylan forms links of the cellulose chain like anhydroglucose. Heuser [17] stated: "The aggregate of chain molecules by which the cellulosic substance may be represented may not consist exclusively of glucose anhydride chains." Sarkar and coworkers [35, 36, 37] have shown, by chemical and chromatographic analysis, that α-cellulose from jute fiber is free from xylan, and jute fiber contains no free xylan or xylan in combination with a hexosan.

It is generally agreed that the physical and mechanical properties of a polymer are a function of its

average molecular weight or D.P., the distribution of the molecular weight about this average, and the molecular architecture or orientation of the molecular chains [4, 11, 16, 41, 42, 43]. Neale emphasized: "Technical interest attaches not so much to the original molecular weight of the natural cellulose, but is chiefly directed to finding a practical answer to the question of how little the cellulose can be depolymerised in the process of purification, so as to preserve its tenacity to the greatest extent" [28].

The usual methods of extraction and purification of cellulose from raw materials involve a more or less degradative action on the cellulose molecule, so that the product obtained has a variable D.P. or molecular weight [29], which is less than that of the native cellulose. The technical product, however, should be as little modified from the original as possible during processing. It should preferably have maximum molecular homogeneity and low copper number, acid value, and uronic carbon dioxide and furfural contents.

The current methods for the isolation of cellulose were critically examined in this laboratory, with special reference to jute, and the results were reported earlier [6, 29]. Further investigations on the evaluation of these methods have since been carried out, based on the determination of the molecular weights of various α-celluloses from viscosity measurements. The results are reported here.

Since viscosity measurement is a very sensitive diagnostic for chain-length degradation, it has been used in the following experiments.

Experimental

Materials and Methods

A medium-quality cotton was cleaned by hand-picking. Average-quality fibers—jute, Mesta, *Urena lobata*, roselle, sunn hemp, true hemp, flax, freed from bark and specks—were obtained in sliver form without batching emulsion, and were cut into short lengths. These were extracted with alcohol-benzene (1:1) for 6 hrs. in a Soxhlet and dried at room temperature.

A sample of ramie was degummed as follows: boiled for 1 hr. with 5% caustic soda solution, washed, boiled for 2.5 hrs. with 5% sodium carbonate solution, washed, and soured. After thorough washing, it was dried at room temperature. Its lignin content was 0.1% (H_2SO_4 method). It was also cut into short lengths.

Oxidizing Treatment

The oxidizing agents which were tried on the fibers were (*a*) chlorine gas (after Cross and Bevan), acting for 45 min. at about 30°C; (*b*) sodium chlorite, 0.7%, and acetic acid, pH 3–4, acting for 2 hrs. on boiling water bath; (*c*) sodium hypochlorite (after Norman and Jenkins), acting twice at neutral and twice at acid pH; and (*d*) chlorine dioxide gas, acting for 72 hrs. at about 30°C. Jute as well as cotton was treated with these chemicals under the optimum conditions of delignification as stated above. Others were delignified with chlorite for comparison among themselves.

For further purification into α-cellulose, the fibers were treated with 17.5% caustic soda solution (*w/w*) at room temperature by the A.C.S. method [32], slightly modified. In some cases 9.3% solution (*w/w*) was also used [35]. Cotton was also purified by Corey and Gray's method [8].

The materials thus obtained were opened and conditioned at standard humidity and temperature, and their moisture contents were determined. Samples for viscosity determination were weighed so as to make the required concentration on the bone-dry weight of the material. Cotton cellulose was taken as the standard for comparison.

Cuprammonium hydroxide and the cellulose solutions were made according to Launer and Wilson [26], with minor alteration, and pyrogallol was used as an antioxidant. Methods of chemical analysis were the same as described previously [6, 38]. Lignin was estimated with 72% sulfuric acid after a preliminary treatment with 5% acid; CO_2 was determined by boiling the material with 12% HCl and absorbing in standard baryta solution; and furfural was determined by distilling with 12% HCl, precipitating the distillate with phloroglucinol in HCl, and weighing as phloroglucide. Acid value was determined by direct titration with *N*/50 NaOH in a medium of salt solution, the material being pretreated with a dilute mineral acid to remove its cationic ash and washed thoroughly. Copper number was estimated with alkaline copper solution according to the Schwalbe-Braidy method after Clibbens and Geake.

Viscosity Determination

This was done according to Clibbens and Geake [7] at 20°C, in the British Cotton Industry Research Association standard capillary viscometers. The values were almost the same as those obtained

with an Ostwald viscometer. For molecular weight of α-cellulose, viscosity was noted for a wide range of concentration—*viz.*, 0.1%–1.0%.

The values for the weight-average molecular weight, and hence D.P., were calculated from Staudinger's equation:

$$\eta_{sp}/C = K_m M,$$

where η_{sp} is the specific viscosity, C is the concentration (in base molar unit), K_m is a constant (5×10^{-4}), and M is the molecular weight.

On plotting η_{sp}/C against C for each sample, curves are obtained, but plotting log η_{sp}/C against C gives straight lines (Figures 1–3), from which the values of intrinsic viscosity,

$$[\eta] = \text{limit } (\eta_{sp}/C)_{C \to 0},$$

may be found by more reliable extrapolation to zero concentration according to Battista [3] or Martin [27]. The concentration in g./dl., adopted in determining intrinsic viscosity, is converted into base molar unit as usual. The results given are the representative values of several observations for each sample, prepared in different lots.

It is pointed out here that Staudinger's equation is a special form of the general equation

$$[\eta] = KM^{\beta},$$

with value of the constant $\beta = 1$, as interpreted by Gee [10].

Intrinsic Viscosity from a Single Viscosity-Concentration Measurement

The intrinsic viscosity $[\eta]$ of a polymer solution is a fundamental constant which is dependent upon the solvent employed as well as upon the conditions of measurement. Various formulas have been suggested [2, 3, 27, 48] to calculate this from viscosity measurement at one concentration only. Calvert and Clibbens [5] have critically examined some of

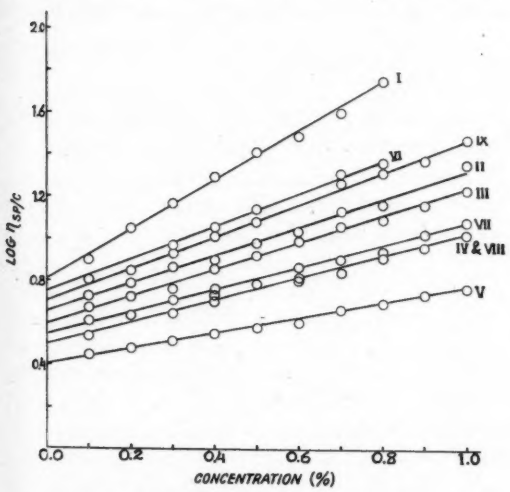

FIG. 1. *Relation between viscosity and concentration of cellulose solutions (Tables III and V).* I—*Purified cotton.* II—*α-Cellulose with 17.5% NaOH, from Cross and Bevan cellulose.* III—*α-Cellulose with 17.5% NaOH, from chlorite cellulose.* IV—*α-Cellulose with 17.5% NaOH, from Norman and Jenkins cellulose.* V—*α-Cellulose with 17.5% NaOH, from chlorine dioxide cellulose.* VI—*α-Cellulose with 9.3% NaOH, from chlorite cellulose.* VII—*α-Cellulose with 9.3% NaOH, from Norman and Jenkins cellulose.* VIII—*α-Cellulose with 9.3% NaOH, from chlorine dioxide cellulose.* IX—*α-Cellulose with 9.3% NaOH, from Cross and Bevan cellulose.*

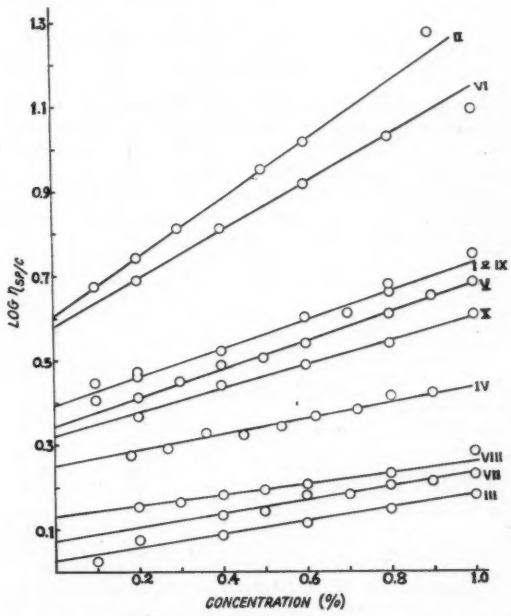

FIG. 2. *Relation between viscosity and concentration of cellulose solutions (Table IV).* I—*Cotton treated with Cl_2.* II—*Cotton treated with $NaClO_2$-AcOH.* III—*Cotton treated with NaClO.* IV—*Cotton treated with ClO_2 gas.* V—*Cotton treated with Cl_2 and further treated with 17.5% NaOH.* VI—*Cotton treated with $NaClO_2$-AcOH and further treated with 17.5% NaOH.* VII—*Cotton treated with NaClO and further treated with 17.5% NaOH.* VIII—*Cotton treated with ClO_2 gas and further treated with 17.5% NaOH.* IX—*Jute α-cellulose treated with $NaClO_2$-AcOH.* X—*Jute α-cellulose treated with $NaClO_2$-AcOH and further treated with 17.5% NaOH.*

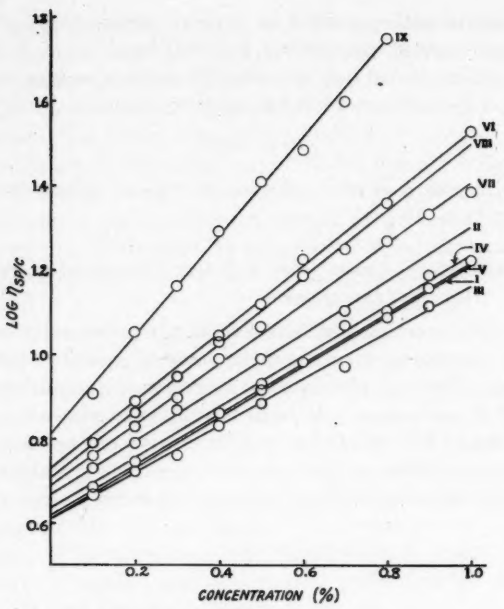

FIG. 3. *Relation between viscosity and concentration of cellulose solutions (Table VII).* I—α-*Cellulose from jute.* II—α-*Cellulose from Mesta.* III—α-*Cellulose from* Urena lobata. IV—α-*Cellulose from roselle.* V—α-*Cellulose from sunn hemp.* VI—α-*Cellulose from true hemp.* VII—α-*Cellulose from flax.* VIII—α-*Cellulose from ramie.* IX—*Purified cotton.*

them—*viz.*,

$$\log \frac{\eta_R - 1}{C} = \log i + miC \qquad (1)$$

(de Jong, Kruyt, and Martin)

$$\eta_R = (1 + iC/N)^N \qquad (2)$$

(Baker)

$$\log i = (p - \log C) - qF_R \qquad (3)$$

(Calvert and Clibbens)

where $p = 0.6$, $q = 1.44$, and $i = [\eta]$. To test how far these formulas fit in with our results, they have been adopted to calculate intrinsic viscosity from our relative viscosity-concentration data. Our prime object, however, was to find true intrinsic viscosity by extrapolation.

Results and Discussion

It may be seen from Table I that the fibers studied fall into two groups: one characterized by comparatively high lignin and hemicellulose contents and with a low yield of α-cellulose; and the other characterized by low lignin and hemicellulose contents and with a high yield of α-cellulose [39].

Effect of Delignification Treatments on the Purity of α-Cellulose from Jute Fiber

Both copper number and acid value are rather high for α-cellulose obtained by the Norman and

TABLE I. ANALYTICAL DATA* FOR LONG FIBERS

Sample	Lignin	CO_2	Furfural	Holocellulose (chlorite)	α-Cellulose	Ash
Jute (*Corchorus olitorius*)	11.48	1.26	9.25	88.8	61.36	0.79
Mesta (*Hibiscus cannabinus*)	8.71	1.37	10.98	88.23	61.62	0.81
Urena lobata	10.25	1.13	10.22	89.62	60.95	0.30
Roselle (*Hibiscus sabdariffa*)	7.16	1.28	10.52	90.03	61.79	0.70
Sunn hemp (*Crotalaria juncea*)	4.20	1.11	2.04	91.09	78.30	1.12
True hemp (*Cannabis sativa*)	5.01	1.24	2.30	82.72	68.61	1.87
Flax	5.53	0.91	2.65	85.86	70.28	1.54
Ramie	0.10	0.73	1.29	—	—	—

* Expressed on 100-g. bone-dry weight of the material.

TABLE II. ANALYTICAL DATA* FOR JUTE α-CELLULOSE†

Treatment of fiber	Copper number (g. Cu/100 g.)	Acid value (NaCl+NaOH) (meq./100 g.)	CO_2 (%)	Furfural (%)	α-Cellulose on jute (%)
Cl_2 gas (after Cross and Bevan)	0.53	2.07	0.26	1.25	60.0
$NaClO_2$-AcOH	0.32	4.08	0.38	1.18	60.4
NaOCl (after Norman and Jenkins)	1.01	2.46	0.30	1.04	61.2
ClO_2 gas	1.13	9.17	0.43	1.02	60.2
Purified cotton	0.09	1.01	0.32	1.02	

* Expressed on bone-dry weight.
† α-Cellulose was prepared with 17.5% NaOH.

Jenkins or the chlorine dioxide method, indicating greater degradation than that shown by the Cross and Bevan as well as the chlorite product, which, nevertheless, give comparable acid values (Table II). Samuelson *et al.* [34] also observed that chlorite bleaching is accompanied by a decrease in copper number and an increase in carboxyl. It was also claimed that breakdown of the cellulose chain can be prevented in chlorine bleach, but oxidation of OH and CHO cannot be prevented [31]. Our results confirm this. It will be seen that the action of chlorine dioxide is the most drastic on cellulose. Since pure cellulose, glucose, and gluconic acid [38] yield small amounts of CO_2, the yield of CO_2 cannot be correlated with the acid values.

The yields of α-cellulose by all the methods studied are practically the same.

Effect of Delignification Treatments on the Molecular Weight of α-Cellulose

In Table III viscosity-concentration data are given for purified cotton and α-cellulose obtained from holocellulose by four different methods. The results are shown graphically in Figure 1. The values of intrinsic viscosity, and hence D.P. or molecular weight, show that the Cross and Bevan or the chlorite product is less degraded than either the Norman and Jenkins or the chlorine dioxide product. The inference drawn from the analytical data for their purity (Table II) is borne out by these results. This finding for jute may be valid for other long vegetable fibers as well.

The over-all quality number (color, strength, D.P.) for cellulose from a kraft pulp was found by Jayme [20] to be higher with chlorine and sodium chlorite treatment than with sodium hypochlorite. Staudinger *et al.* [44] also observed from D.P. measurements of α-cellulose from chlorite holocellulose of wood that there was hardly any degradation. Other investigators [18, 21, 23, 30, 50] also claimed the superiority of chlorine gas and chlorite as reagents for cellulose extraction. Hatch [13] elaborated the advantages of the Cross and Bevan method for cellulose over hypochlorite oxidation.

Effect of Delignification Treatments on Pure Cellulose

To gain information on the extent of degradation or depolymerization of cellulose due to these methods, the delignification treatments were applied to extracted (defatted) cotton, and the molecular weights of various residues were determined

TABLE III. VISCOSITY–CONCENTRATION DATA FOR PURIFIED COTTON AND JUTE α–CELLULOSE FROM VARIOUS SOURCES

Concentration (g./100 ml.)	η_{sp}	η_{sp}/C	log η_{sp}/C	Intrinsic viscosity $[\eta]$ (dl./g.)	D.P.	Molecular weight
			Purified cotton			
0.1	0.801	8.01	0.90			
0.2	2.254	11.27	1.05			
0.3	4.377	14.59	1.16			
				6.50	1300	210,600
0.4	7.820	19.55	1.29			
0.5	12.780	25.56	1.41			
0.6	18.330	30.55	1.49			
0.7	27.870	39.81	1.60			
0.8	44.770	55.96	1.75			
			α-Cellulose from Cross and Bevan cellulose			
0.1	0.531	5.31	0.73			
0.2	1.214	6.07	0.78			
0.3	2.182	7.27	0.86			
0.4	3.160	7.90	0.90	4.48	896	145,152
0.5	4.740	9.48	0.98			
0.6	6.534	10.89	1.04			
0.7	9.450	13.50	1.13			
0.8	11.592	14.49	1.16			
0.9	14.283	15.87	1.20			
1.0	22.560	22.56	1.35			
			α-Cellulose from chlorite cellulose			
0.1	0.467	4.67	0.67			
0.2	1.050	5.25	0.72			
0.3	1.722	5.74	0.76			
0.4	2.884	7.21	0.86	4.00	800	129,600
0.5	4.140	8.28	0.92			
0.6	5.826	9.71	0.99			
0.7	8.022	11.46	1.06			
0.8	9.792	12.24	1.09			
0.9	13.014	14.46	1.16			
1.0	17.080	17.08	1.23			
			α-Cellulose from Norman and Jenkins cellulose			
0.1	0.343	3.43	0.54			
0.2	0.852	4.26	0.63			
0.3	1.302	4.34	0.64			
0.4	2.160	5.40	0.73	3.16	632	102,384
0.5	2.785	5.57	0.75			
0.6	3.906	6.51	0.81			
0.7	4.851	6.93	0.84			
0.8	6.928	8.66	0.94			
0.9	8.280	9.20	0.96			
1.0	10.420	10.42	1.02			
			α-Cellulose from chlorine dioxide cellulose			
0.1	0.278	2.78	0.44			
0.2	0.596	2.98	0.47			
0.3	0.969	3.23	0.51			
0.4	1.404	3.51	0.55	2.53	506	81,972
0.5	1.885	3.77	0.58			
0.6	2.370	3.95	0.60			
0.7	3.185	4.55	0.66			
0.8	3.936	4.92	0.69			
0.9	4.887	5.43	0.73			
1.0	5.820	5.82	0.76			

before and after treatment with 17.5% caustic soda. The results are given in Table IV and Figure 2.

These results reveal that the common bleaching agents attack pure cellulose to a considerable extent, apparently more severely than lignocelluloses. Lignin or small-molecule products present with cellulose seem to be more susceptible to oxidation and shield the cellulose chain. From heterogeneous acid hydrolysis of jute and its chlorite holocellulose, we have found that their hydrolysis-time curves are very similar. Moreover, the curve for jute is not less steep than that for holocellulose. This apparently indicates that lignin offers very little protection to cellulose from hydrolysis. Hemicelluloses are more susceptible to oxidation than α-cellulose, so they serve to protect the latter at least partially by being more rapidly oxidized. A similar observation was made by others [12, 25, 33] with sulfite or kraft pulps which were less affected than cotton by chlorite or strong alkali treatment. Hatch [13] remarked that noncellulosic impurities associated with cellulose may be selectively oxidized, and, as they disappear, the danger of degradation of cellulose increases.

The Cross and Bevan and the chlorite methods are again the least degradative. It is pointed out that in the case of cotton cellulose, depolymerization is less with chlorite than chlorine, whereas in the case of α-cellulose, it is less in chlorinated jute than in jute treated with chlorite. Similar results were recently obtained by other workers [47] using chlorine and chlorite on wood and cotton linters.

A marked fall in the D.P. of cotton cellulose by treatment with 0.6% ClO_2 solution was observed by Staudinger *et al.* [45]. Interesting observations on the attack upon cotton cellulose by sodium chlorite, hypochlorite, chlorine dioxide, etc., have recently been reported [25, 53]. The D.P. of cotton was markedly lowered by treatment with 1% chlorite at 90°C and 0.5% ClO_2 solution with

TABLE IV. EFFECT OF THE DELIGNIFICATION TREATMENTS ON PURE CELLULOSE

Sample and treatment	Intrinsic viscosity [η] (dl./g.)	D.P.	Molecular weight
Purified cotton	6.50	1300	210,600
Cotton treated with Cl_2 after Cross and Bevan	2.46	492	79,704
Do. further treated with 17.5% NaOH	2.21	442	71,604
Cotton treated with $NaClO_2$-AcOH	4.03	806	130,572
Do. further treated with 17.5% NaOH	3.80	760	123,120
Jute α-cellulose (from chlorite holocellulose with 9.3% NaOH)	5.75	1150	186,300
Do. treated with $NaClO_2$-AcOH	2.43	486	78,732
Do. further treated with 17.5% NaOH	2.11	422	68,364
Jute α-cellulose (from chlorite holocellulose with 9.3% NaOH) treated with H_2O_2 at pH 10–11*	2.90	580	93,960
Cotton treated with NaClO after Norman and Jenkins	1.05	210	34,020
Do. further treated with 17.5% NaOH	1.18	236	38,232
Cotton treated with ClO_2 gas	1.80	360	58,320
Do. further treated with 17.5% NaOH	1.35	270	43,740

* H_2O_2 in 1 vol. strength, containing 1% sodium silicate and 0.5% sodium phosphate, temperature 65°–70°C, is apparently less degradative to α-cellulose. This supports the work of Kaufmann [22].

TABLE V. MOLECULAR SIZE OF α-CELLULOSE OBTAINED WITH DIFFERENT CONCENTRATIONS OF ALKALI

Source of α-cellulose	Concentration of NaOH (%)	Intrinsic viscosity [η] (dl./g.)	D.P.
Cotton treated with Cl_2 (after Cross and Bevan)	17.5	2.21	442
	9.3	2.48	496
Jute treated (after Cross and Bevan)	17.5	4.48	896
	9.3	5.13	1026
Cotton treated with $NaClO_2$-AcOH	17.5	3.80	760
	9.3	3.80	760
Jute treated with $NaClO_2$-AcOH	17.5	4.00	800
	9.3	5.75	1150
Cotton treated with NaOCl (after Norman and Jenkins)	17.5	1.18	236
	9.3	1.18	236
Jute treated with NaOCl (after Norman and Jenkins)	17.5	3.16	632
	9.3	3.60	720
Cotton treated with ClO_2 gas	17.5	1.35	270
	9.3	1.33	266
Jute treated with ClO_2 gas	17.5	2.53	506
	9.3	3.13	626

decreasing pH. A rise in copper number and acid value with the ClO_2 cellulose was also observed. Our results corroborate these.

The wide difference in molecular weight between the products from the Cross and Bevan and the Norman and Jenkins procedures, although chlorine is apparently the active oxidant in both cases, may be explained by the following facts. Gaseous chlorine is used in the former case, whereas nascent chlorine (much more reactive) liberated from hypochlorite by sulfuric acid is employed in the latter case. Further, it is well known that the danger zone of hypochlorite bleach liquor occurs near the neutral point. In the Norman and Jenkins method, neutral as well as acid hypochlorite is used repeatedly, with obvious results. As expected, alkali treatment after oxidation brought about a further degradation of the cellulose molecule in a majority of the cases.

Effect of the Concentration of Caustic Soda on the Resulting α-Cellulose

The results are given in Table V and are shown graphically in Figure 1. It may be seen that a less degraded α-cellulose is generally obtained from jute when a lower concentration of caustic soda is employed for its extraction. Using 8%–10% caustic soda, Staudinger *et al.* [44] extracted from chlorite holocellulose an α-cellulose, free from xylan, which was hardly degraded.

Effect of Grinding on the Molecular Weight of Cellulose

Samples of α-cellulose from jute and purified cotton were pulverized in a Christy and Norris Laboratory Mill fitted with an 8-mesh screen, and their molecular weights were determined as usual. The results are shown in Table VI.

TABLE VI. CHANGE OF MOLECULAR SIZE OF CELLULOSE ON DISINTEGRATION

Sample	Intrinsic viscosity [η] (dl./g.)	D.P.	Molecular weight
Jute α-cellulose No. 1	5.72	1144	185,328
Do. pulverized	5.00	1000	162,000
Jute α-cellulose No. 2	5.30	1060	171,720
Do. pulverized	4.79	958	155,196
Purified cotton (Cambodia CO_2)*	8.71	1742	282,204
Do. pulverized	6.84	1368	221,616

*Received from Director, Technological Laboratory, Indian Central Cotton Committee, Bombay, India.

It is evident that depolymerization occurs on mechanical disintegration. This was also observed by other workers [14, 19, 24, 51, 52]. The D.P. values of wood pulp, cotton, and ramie were found to be gradually reduced with time by dry grinding. It is supposed that the energy necessary for the rupture of the cellulose chain is derived mainly from mechanical action. The smaller fall in D.P. of α-cellulose than of cotton may be explained by the fact that α-cellulose even before dry grinding had already been depolymerized by mechanical kneading during mercerization as well as by accelerated oxidation. This view is supported by the work of other investigators [1, 9, 21, 49] who claimed that α-cellulose is degraded during its extraction from holocellulose.

Molecular Size of α-Cellulose from Various Sources

α-Cellulose was obtained with 17.5% caustic soda solution from the chlorite holocellulose of long fibers, and the molecular weights were determined. The results are shown in Table VII and Figure 3.

Of the bast fibers, the high-lignin-content group (first four of Table VII) has generally a lower D.P. than the low-lignin-content group (next four of Table VII). It is evident from Figure 3 that curves VI–IX show greater divergence from the others with rise of concentration. It is probable that this is simply a reflection of the larger slopes of the curves for material of higher D.P., which in turn is indicative of the increased interaction of the longer molecular chains at higher concentrations. It is not unlikely that cellulose in the second group is more susceptible to depolymerization processes. But, unless the D.P. of the materials is previously known, it is difficult to draw reliable conclusions about their susceptibility to depolymerization. It has recently

TABLE VII. MOLECULAR SIZE OF α-CELLULOSE FROM VARIOUS SOURCES

Source	Intrinsic viscosity [η] (dl./g.)	D.P.	Molecular weight
Jute	4.00	800	129,600
Mesta	4.68	936	151,632
Urena lobata	4.07	814	131,868
Roselle	4.40	880	142,560
Sunn hemp	4.17	834	135,108
True hemp	5.37	1074	173,988
Flax	4.90	980	158,760
Ramie	5.01	1002	162,324
Purified cotton	6.50	1300	210,600

TABLE VIII. INTRINSIC VISCOSITY OF JUTE α-CELLULOSE FROM CHLORITE CELLULOSE, CALCULATED IN DIFFERENT WAYS

Concentration (g./100 ml.)	Relative viscosity	Intrinsic viscosity [η] calculated from: de Jong, Kruyt, and Martin equation (m=0.14) (1)	Baker equation (N=3.5) (2)	Calvert and Clibbens equation (3)
0	—	←———	4.00*	———→
0.1	1.467	4.07	4.03	4.15
0.2	2.050	4.06	3.97	3.95
0.3	2.723	3.92	3.86	3.93
0.4	3.883	4.15	4.14	4.24
0.5	5.139	4.18	4.17	4.18
		Mean 4.08	Mean 4.03	
0.6	6.848	4.28	4.28	4.09
0.7	9.027	4.31	4.38	3.94
				Mean 4.07
0.8	10.795	4.14	4.25	3.66
0.9	14.020	4.23	4.38	3.49
1.0	18.080	4.27	4.50	3.31

* True value of intrinsic viscosity, found by extrapolation to zero concentration.

been found in this laboratory that the composition of hemicellulose in the two groups of fibers is fundamentally different. This will be reported shortly.

The D.P. values of some of the fiber celluloses given by Staudinger and Feuerstein [46] are very much higher. It should be remembered that the D.P. of a product is dependent upon its method of determination.

Adaptability of Some Equations to Our Viscosity-Concentration Data, to Derive Intrinsic Viscosity

It is evident from Table VIII that all three equations give values very near the extrapolated one: the first two, in a range of relative viscosities up to 5; and the third, in a range up to 9. But such a value was seldom obtained with the Schulz and Blaschke Equation [40].

The viscosity of a 0.5% solution is ordinarily used in the routine characterization of cellulose materials. Therefore, the true values of intrinsic viscosity (found by extrapolation) of sixteen cellulose preparations, together with the corresponding values of relative viscosity, $\eta_{0.5}$, of 0.5% solution, are tabulated in Table IX for comparison.

On plotting the data, a linear relationship (with a high correlation coefficient, +0.993) was found to

TABLE IX. RELATION BETWEEN INTRINSIC VISCOSITY AND RELATIVE VISCOSITY, $\eta_{0.5}$

Sample and treatment	Intrinsic viscosity [η]	Relative viscosity $\eta_{0.5}=(1+\eta_{sp})$
Purified cotton	6.50	13.78
Cotton purified and pulverized	5.90	10.47
Cotton treated after Cross and Bevan	2.46	2.69
Cotton treated with $NaClO_2$-AcOH	4.03	5.44
Cotton treated with NaOCl after Norman and Jenkins	1.05	1.66
Do. further treated with 17.5% NaOH	1.18	1.69
Cotton treated with ClO_2 and further treated with 17.5% NaOH	1.35	1.78
Jute treated after Cross and Bevan and further treated with 17.5% NaOH	4.48	5.74
Jute treated after Cross and Bevan and further treated with 9.3% NaOH	5.13	7.03
Jute treated with $NaClO_2$-AcOH and further treated with 17.5% NaOH	4.00	5.14
Jute treated with $NaClO_2$-AcOH and further treated with 9.3% NaOH	5.75	7.82
Jute treated after Norman and Jenkins and further treated with 17.5% NaOH	3.16	3.78
Jute treated after Norman and Jenkins and further treated with 9.3% NaOH	3.60	3.99
Jute treated with ClO_2 and further treated with 17.5% NaOH	2.53	2.59
Jute α-cellulose treated with $NaClO_2$-AcOH	2.43	2.81
Jute α-cellulose treated with H_2O_2	2.90	2.99

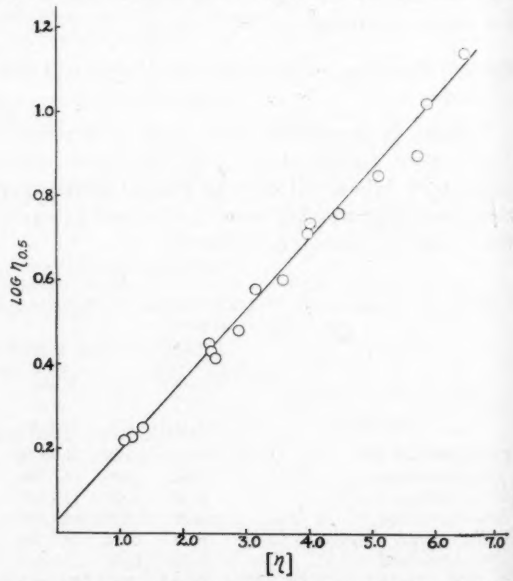

FIG. 4. *Relation between relative viscosity of 0.5% solution and intrinsic viscosity of cellulose (Table IX).*

TABLE X. RELATIVE VISCOSITY AT 0.5% CONCENTRATION AND INTRINSIC VISCOSITY OF α-CELLULOSE FROM VARIOUS SOURCES

Source	Relative viscosity $\eta_{0.5}$	Intrinsic viscosity		Deviation from true value (%)
		True	Calculated	
Mesta	5.909	4.68	4.52	−3.4
Urena lobata	4.815	4.07	3.98	−2.2
Roselle	5.296	4.40	4.23	−3.9
Sunn hemp	4.935	4.17	4.04	−3.1
True hemp	7.590	5.37	5.19	−3.4
Flax	6.844	4.90	4.91	+0.2
Ramie	7.605	5.01	5.19	+3.6

exist between the relative viscosity function, log $\eta_{0.5}$, and intrinsic viscosity $[\eta]$, and hence D.P. or molecular weight. This is shown graphically in Figure 4 and may be represented by the following equation:

$$\log \eta_{0.5} = 0.03286 + 0.16348\,[\eta],$$

the error of estimate being ± 0.0343. Similar equations, although not identical in form, were reported by several workers, including Battista [3] and Tripp *et al.* [48].

The accuracy of this equation was tested by calculating the intrinsic viscosities of α-celluloses from various other sources (not utilized in deriving the equation) from the relative viscosity data of their 0.5% solutions. The calculated and true values are given in Table X. It may be seen that the calculated values are within the limits of experimental error.

Further work on cellulose with a wider range of intrinsic viscosity is necessary in order to test the validity of this equation. It is, however, recorded here in the hope that it may help in orienting further research.

Summary

Four common methods for the extraction of cellulose from long vegetable fibers, with special reference to jute, have been critically studied. Jute and cotton were treated, under optimum conditions of delignification, with chlorine gas (after Cross and Bevan), sodium chlorite-acetic acid, sodium hypochlorite (after Norman and Jenkins), and chlorine dioxide gas. Seven other long fibers were treated with the chlorite. The products were further purified to α-cellulose with 17.5% and 9.3% caustic soda solution. The materials thus obtained were analyzed for chemical purity, and their molecular weights were determined by the measurement of viscosity in cuprammonium hydroxide solution.

Cellulose is depolymerized to different extents by all of the methods, pure cellulose being depolymerized to a much greater extent than lignocelluloses. Lignin and hemicellulose apparently protect α-cellulose. The Cross and Bevan method gives the least degraded product from a lignocellulose, whereas the chlorite treatment seems to be the best for cotton. Generally, the use of a higher concentration of alkali than 9.3% for mercerization or disintegration of the material in a laboratory mill causes further degradation.

The molecular weight data of α-cellulose from chlorite holocellulose give an indication that α-cellulose may be more highly polymerized in fibers with low lignin content than in fibers with high lignin content.

From analysis of the viscosity-concentration data for sixteen samples of jute and cotton cellulose, an equation has been suggested to calculate intrinsic viscosity from relative viscosity of a cellulose solution in cuprammonium at 0.5% concentration. The accuracy of this equation was tested by calculating intrinsic viscosity of α-celluloses from the other long fibers and comparing the calculated values with the corresponding true values, found by extrapolation to zero concentration. The agreement is fairly satisfactory. The adaptability of some other equations to our data has also been examined.

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Deviations in Instrumental Evaluation of the Physical Properties of Cotton

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A SERIES of interlaboratory check tests of the physical properties of cotton, as evaluated with instruments, was conducted during 1951 and 1952 by a Task Group of Committee D-13 of the American Society for Testing Materials. The test program was designed to compare the combined performance of operators and instruments and not to compare the physical properties of cottons. One of the objectives was to obtain data from which indications of precision in testing in a laboratory and between laboratories could be derived by statistical analysis of the results of testing with the Fibrograph,† Pressley, and Micronaire for length, strength, and fineness, respectively [3].

Nine lots of cotton were obtained and used for the preparation of check samples. Each lot was blended by the Southern Regional Research Laboratory to provide the maximum uniformity within each of them by use of textile mill equipment. In each case the cotton was subjected to opening and picking, carding, and recarding. Conventional speeds, settings, and weights per yard were used. In preparing the samples for the 1951 tests, the sliver from the second carding was packaged in ¼-lb. quantities to provide individual samples for distribution. The samples used in the 1952 test program were packaged in ½-lb. quantities from the card web.

The Suter-Webb array, weight fineness, and maturity of each blended lot of cotton, determined by the Southern Regional Research Laboratory, are given in Table I.

Ten packages were selected at random from each of the nine lots of blended and packaged cottons prepared for distribution. They were tested at the Southern Regional Research Laboratory for length, strength, and fineness by use of the Fibrograph, Pressley, and Micronaire instruments, respectively. This was done to obtain information on the uniformity of the blending and subsampling. Ten tests were made for each property on each of the ten samples randomly selected from each lot of cotton. The test results are summarized in Table II. The analysis of the test data obtained with the Fibrograph was limited to the upper-half mean length. The standard deviations are for the average values of each of the ten samples selected for test in each instance. Recognizing that the variations in the results include both those in blending and sampling and those inherent

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† Mention of the names of firms or trade products does not imply that they are endorsed or recommended by the U. S. Department of Agriculture over other firms or similar products not mentioned.

TABLE I. SUTER-WEBB ARRAY, WEIGHT FINENESS, AND MATURITY OF CHECK COTTONS

No.	Variety	Upper quartile length (in.)	Mean length (in.)	Coefficient of variation (%)	Weight fineness (μg./in.)	Maturity (NaOH) (%)
1951 Check Samples						
1	Rowden	1.02	0.80	34	5.4	86
2	Deltapine	1.23	1.06	26	4.4	83
3	Dixie Triumph	1.06	0.86	30	4.6	74
4	Sealand	1.46	1.10	40	3.4	72
5	Acala 1-23-11-3	1.16	0.98	27	4.4	83
1952 Check Samples						
6	Rowden	0.98	0.78	34	5.4	84
7	Stoneville 2B	1.14	0.89	36	4.2	82
8	Acala 1517	1.25	1.04	28	4.2	86
9	Wilds 13	1.32	1.04	38	3.8	80

in instrument and operator performance, it is thought that the samples were very well blended in each instance.

Ten check samples, two each of cottons nos. 1–5 inclusive, were sent one at a time at 2-week intervals in 1951 to 83 laboratories. Four check samples, one each of nos. 6–9 inclusive, were sent at one mailing in 1952 to each of 142 laboratories. The participants in the testing program of 1951 were requested to perform the tests according to the procedures regularly in use in their respective laboratories. However, in 1952 they were requested to check all balances used with standard weights and to use either specific A.S.T.M. or instrument manufacturers' methods.

The results of the interlaboratory tests are summarized in Table III. It was observed generally that laboratories having an average high or low value of test consistently reported high or low values correspondingly for all individual check samples. A few exceptions were observed. The results of both years' check tests indicate the need of adherence to standardized test procedures and the use of check cottons of known properties to check both instrument and operator performance to assure a reasonably even level of testing in a laboratory or between laboratories.

The results provided two groups of symmetrical data for analysis of precision attained within each of the several laboratories and between the laboratories. The first group consisted of the data of the Southern Regional Research Laboratory on ten randomly selected samples of each of the nine lots of blended cotton. It comprised ten tests with each instrument on each of these samples. The second group consisted of the data reported by selected laboratories, each testing two samples of each lot of cotton in 1951 and one sample of each in 1952. The criteria by which these laboratories were selected from those participating in the check sample testing were: (*1*) participation in both 1951 and 1952 test programs and reporting on all samples; (*2*) use of standard atmosphere, 65% R.H. and 70°F; and (*3*) performance of five or more individual tests on each sample. The number of laboratories meeting these requirements in using the Fibrograph, Pressley, and Micronaire instruments were 12, 25, and 11, respectively. A summary of the results reported by these laboratories is given in Table IV. Individual test values were reported in 1951, while only mean values were requested of the participants in 1952.

The individual test data obtained with the Fibrograph and Pressley instruments on each of the ten samples of each of the nine check cottons tested at the Southern Regional Research Laboratory were subjected to analysis of variance [4].

The within- and between-sample variance for the data of the selected laboratories were estimated by the method of ranges [2] in obtaining over-all standard deviations.

Fibrograph Upper-Half Mean Length

The results of the analysis of variance of the test data for the ten randomly selected samples of each

TABLE II. SUMMARY OF EVALUATION OF 10 RANDOMLY PICKED SAMPLES OF EACH CHECK COTTON

	Fibrograph U.H.M. (in.)*		Pressley index		Micronaire value†	
No.	Mean	S.D.‡	Mean	S.D.	Mean	S.D.
1	0.952	0.010	7.55	0.11	5.32	0.06
2	1.158	0.005	7.27	0.06	4.50	0.07
3	0.992	0.007	6.76	0.08	4.36	0.04
4	1.324	0.006	7.94	0.07	3.17	0.04
5	1.081	0.011	6.57	0.08	4.53	0.05
6	0.932	0.007	7.37	0.07	5.34	0.04
7	1.049	0.006	7.37	0.06	4.48	0.03
8	1.153	0.006	7.72	0.09	4.57	0.02
9	1.261	0.006	8.30	0.08	3.94	0.01

* Upper-half mean length.
† Lineal scale for 1951 and curvilinear scale for 1952 tests.
‡ Standard deviation.

TABLE III. SUMMARY OF INTERLABORATORY CHECK SAMPLE TEST RESULTS

	Fibrograph U.H.M. (in.)		Pressley index		Micronaire value*	
No.	Mean	S.D.	Mean	S.D.	Mean	S.D.
1951 Test Samples†						
1	0.946	0.029	7.46	0.35	5.30	0.22
2	1.166	0.033	7.18	0.34	4.55	0.23
3	0.993	0.031	6.86	0.32	4.37	0.23
4	1.301	0.056	7.90	0.38	3.26	0.24
5	1.091	0.029	6.75	0.34	4.56	0.21
1952 Test Samples‡						
6	0.940	0.029	7.50	0.38	5.34	0.25
7	1.052	0.037	7.50	0.37	4.45	0.23
8	1.151	0.039	7.74	0.36	4.58	0.20
9	1.257	0.039	8.28	0.40	3.96	0.18

* Lineal scale for 1951 and curvilinear scale for 1952 tests.
† The number of participating laboratories were 55, 69, and 55 for the Fibrograph, Pressley, and Micronaire tests, respectively. The number of reports for each test were twice or very nearly twice the number of participants.
‡ The number of participating laboratories and reports for each test were 66, 86, and 100 for the Fibrograph, Pressley, and Micronaire tests, respectively.

of the nine check cottons are tabulated in Table V. The variation in test results indicated by the standard deviations includes any residual nonuniformity in the blended cottons and fluctuations in both operator and instrument performance.

The within-sample estimated over-all standard deviation for all the tests on the randomly selected samples of the nine check cottons was found to be 0.017 in. A similar estimate by the method of ranges [2] for the results of the twelve selected laboratories testing two samples each of check cottons nos. 1–5 inclusive was 0.012 in.

The between-sample estimated over-all standard deviation of the mean values for all of the randomly selected samples was found to be 0.008 in. A similar estimate by the method of ranges for the mean values reported by the twelve selected laboratories was 0.018 in. The estimated values for samples nos. 1–5 and nos. 6–9 were 0.023 and 0.013 in., respectively. However, if sample no. 4 (Sealand) is omitted from consideration, the estimated standard deviation for samples nos. 1, 2, 3, and 5 was found to be 0.018 in. The fact that samples nos. 6–9 were generally tested on one day may account for the low standard deviation observed. Samples nos. 1–5 were tested at intervals over a period of 5 months.

The number of tests or observations required to attain the specified precision in testing a single sample for length with a Fibrograph has been estimated by accepted methods [1] and tabulated in Table VI. The calculations were based on an assumed within-sample standard deviation of 0.012 in., which was observed as the over-all value for the performance of the twelve selected laboratories in testing check cottons nos. 1–5.

The over-all standard deviation of the mean results for upper-half mean length reported by the twelve selected laboratories in testing the nine check cottons was calculated from the standard deviations given in Table IV. It was found to be 0.028 in. It may be assumed that this value could be reduced if the laboratories were all to use samples of the same lot of check cottons in maintaining an even level of testing.

TABLE IV. SUMMARY OF INTERLABORATORY TEST RESULTS SELECTED FOR STATISTICAL ANALYSIS

	Fibrograph U.H.M. (in.)		Pressley index		Micronaire value*	
No.	Mean	S.D.	Mean	S.D.	Mean	S.D.
1	0.947	0.015	7.45	0.32	5.36	0.13
2	1.173	0.022	7.20	0.36	4.62	0.14
3	1.000	0.018	6.94	0.31	4.41	0.20
4	1.309	0.038	8.01	0.34	3.30	0.21
5	1.098	0.028	6.79	0.30	4.58	0.21
6	0.939	0.024	7.48	0.31	5.30	0.09
7	1.050	0.034	7.49	0.28	4.43	0.10
8	1.154	0.023	7.74	0.40	4.55	0.09
9	1.259	0.038	8.30	0.43	3.91	0.12

* Lineal scale for 1951 and curvilinear scale for 1952 tests.

TABLE V. ANALYSIS OF VARIANCE OF FIBROGRAPH UPPER-HALF MEAN LENGTH FOR 10 RANDOMLY PICKED SAMPLES OF EACH CHECK COTTON

		Standard deviation		Coefficient of variation	
No.	*F* value	Within samples (in.)	Between samples (in.)	Within samples (%)	Between samples (%)
1	1.79	0.016	0.010	1.7	1.04
2	0.78	0.018	0.005	1.7	0.44
3	1.67	0.017	0.007	1.7	0.71
4	0.52	0.028	0.006	2.1	0.49
5	3.66*	0.018	0.011	1.7	1.01
6	2.23*	0.016	0.007	1.7	0.78
7	2.69*	0.012	0.006	1.1	0.59
8	2.32*	0.012	0.006	1.0	0.50
9	2.13*	0.014	0.006	1.1	0.52

* Significant: $F(P = 0.95) = 1.98$ and $F(P = 0.99) = 2.62$.

Pressley Index

The analysis of variance [4] of the test data for the randomly selected ten samples of each check cotton is given in Table VII. As in the case of the Fibrograph data, the variation observed within samples is a combination of residual nonuniformity and fluctuation in both operator and instrument performance.

The estimated within-sample over-all standard deviation for all tests on the ten randomly selected samples of each of the nine check cottons was

TABLE VI. TESTS REQUIRED TO ATTAIN SPECIFIED PRECISION IN MEASUREMENT OF UPPER-HALF MEAN LENGTH WITH A FIBROGRAPH*

Precision (in.)	Confidence limits (approx.) (%)	Tests or observations required	Range in individual observations (in.)		
			Max.	Min.	Mean
±0.03	95	3	0.05	0	0.02
±0.03	99	5	0.06	0	0.03
±0.01	95	10	0.07	0.01	0.04
±0.01	99	15	0.07	0.01	0.04

* Based on over-all within sample standard deviation of 0.012 in.

calculated as 0.19 Pressley index units. A similar estimate by the method of ranges [2] for the results of the 25 selected laboratories testing two samples each of check cottons nos. 1–5 inclusive gave standard deviations ranging from 0.08 to 0.42 Pressley index units. The pooled value was 0.20, which compared very favorably with the pooled value of 0.19 observed for the randomly selected samples of the cottons tested at the Southern Regional Research Laboratory.

The between-sample over-all standard deviation of the mean values for all of the randomly selected samples was 0.08 Pressley index units. A similar estimate by the method of ranges for the mean values reported by the 25 selected laboratories was 0.20 Pressley index units. The estimated values for check cottons nos. 1–5 and nos. 6–9 were 0.23 and 0.14, respectively. The larger value for check cottons nos. 1–5 may be attributed to the observation that the check samples of each were tested by the participating laboratories at regular intervals instead of at one time as in the case of check cottons nos. 6–9.

The number of tests or observations required to attain the specified precision in testing a single sample for strength with the Pressley instrument has been estimated [1] and tabulated in Table VIII. The calculations are based on an assumed within-sample standard deviation of 0.20 Pressley index units observed as the over-all value for the performance of the 25 selected laboratories in testing check cottons nos. 1–5.

The over-all standard deviation of the Pressley indices reported by the 25 laboratories in testing the nine check cottons was calculated from the standard deviations given in Table IV. It was found to be 0.34. As in the case of length measurement with the Fibrograph, it is assumed that use of check cottons to maintain an even level of test should result in a reduced deviation in results between laboratories.

TABLE VII. ANALYSIS OF VARIANCE OF PRESSLEY INDICES OF STRENGTH FOR 10 RANDOMLY PICKED SAMPLES OF EACH CHECK COTTON

No.	F value	Standard deviation (Pressley units) Within samples	Between samples	Coefficient of variation Within samples (%)	Between samples (%)
1	2.35*	0.22	0.11	3.0	1.4
2	0.95	0.19	0.06	2.7	0.8
3	2.50*	0.17	0.08	2.5	1.3
4	1.43	0.20	0.08	2.5	1.0
5	2.21*	0.18	0.08	2.5	1.3
6	1.53	0.18	0.07	2.4	0.9
7	1.24	0.18	0.06	2.4	0.9
8	2.15*	0.19	0.09	2.4	1.1
9	1.86	0.21	0.09	2.5	1.1

* Significant: $F(P = 0.95) = 1.98$ and $F(P = 0.99) = 2.62$.

Micronaire Values

By comparing the standard deviations for Micronaire values of fineness reported by all participants (Table III) and the eleven selected participants (Table IV) with standard deviations of the mean values obtained in a single laboratory on the ten randomly selected samples of each of the nine check cottons (Table II), some wide variations are noted. The over-all standard deviations for these three sets of test data are 0.22, 0.15, and 0.04, respectively. These values lead to the assumption that fixed biases contribute to the variations in results reported by the different laboratories. The standard deviation found for the selected laboratories maintaining the standard atmosphere and making five or more tests per sample was significantly lower than the standard deviation for the results from all participating laboratories, yet more than three times as large as the value for the one laboratory observing recommended test conditions and testing the ten randomly selected samples of each of the nine check cottons. The precision of a laboratory's mean Micronaire value, in comparison with the population mean value, may be estimated from the above figures. For the selected laboratories with a standard deviation of the mean value of 0.15, a precision of approximately $\pm$ 0.3 units may be had at the 95% confidence level.

Summary

Through interlaboratory testing of samples of nine lots of blended cottons, data have been obtained to

TABLE VIII. TESTS REQUIRED TO ATTAIN SPECIFIED PRECISION IN MEASUREMENT OF PRESSLEY INDEX OF STRENGTH *

Precision (Pressley units)	Confidence limits (approx.) (%)	Tests or observations required	Range in individual observations, units Max.	Min.	Mean
±0.2	95	6	1.0	0	0.5
±0.2	99	12	1.1	0.2	0.7
±0.1	95	20	1.2	0.3	0.7
±0.1	99	30	1.3	0.4	0.8

* Based on over-all within-sample standard deviation of 0.20 Pressley units.

indicate deviations in test results as presently obtained in and between laboratories in the use of the Fibrograph, Pressley, and Micronaire instruments for the evaluation of length, strength, and fineness, respectively.

The precision attained by a group of laboratories maintaining the standard atmosphere and making five or more tests per sample is indicated by a standard deviation of 0.012 in. Fibrograph upper-half mean length and of 0.20 units in Pressley index of strength for individual observations. Similarly, the precision of mean values for the laboratories meeting the above criteria is indicated by a standard deviation of 0.018 in. Fibrograph upper-half mean length, 0.20 units Pressley index of strength, and 0.15 units Micronaire fineness. The between-sample mean values of the standard deviations observed for a single laboratory testing ten samples of each of the nine check cottons were 0.008, 0.08, and 0.04, respectively.

It may be postulated that the agreement or precision between laboratories may be greatly improved by adherence to a single source of test procedures, use of standard atmospheric conditions of 65% R.H. and 70°F, and general use of standardized check cottons to maintain a uniform level of testing.

Deviations or results in testing clasers' samples are expected to be larger than those observed in this study unless the blending and subsampling are done as thoroughly as in preparing these check samples.

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(Manuscript received July 6, 1953.)

Letters to the Editor

Short communications in the form of Letters to the Editor are intended to provide prompt publication of significant new research results and to permit an exchange of views on papers previously published in the JOURNAL. These communications are not submitted to formal review as are research papers, and the editors do not assume any share of the author's responsibility for the information given or the opinions expressed. When work previously published in the JOURNAL is the subject of critical comment, the authors of the original paper are given an opportunity to submit a reply, which will be published concurrently when possible.

The Crimping and Heterogeneity of Keratin Fibers

THE UNIVERSITY
Department of Textile Industries
Leeds, England
October 17, 1953

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

In their papers on the above subject, Horio and Kondo [1] and Mercer [2] made no reference to the chemical treatments which have long been used to straighten fibers in the manufacture of furs. These treatments are based on the assumption that crimp is associated with strained disulfide bonds, and that the latter will react preferentially with, for example, oxidizing agents. The possibilities of such treatments are illustrated by the following data, which were obtained by treating merino wool fibers with mixtures of sulfuric acid and potassium dichromate for 1 hr. at 41°C. A measure of the extent to which crimp is removed was obtained by measuring the air-dry length of each fiber after it had been drawn taut, l_t, as well as when just sufficient force was applied to the horizontal fiber to give uniform distribution about an imaginary line joining the supports, l_c. Similar measurements of the uncrimped and crimped lengths were made after oxidation. The percentage reduction in crimp is defined as

$$\left[1 - \frac{l_{t_2} - l_{c_2}}{l_{t_1} - l_{c_1}}\right] \cdot 100.$$

The solutions used were prepared by mixing 10 ml of 0.4, 4.0, or 8.0*N* sulfuric acid with 50 ml. of 0.2%, 1.0%, or 5.0% solution of potassium dichromate. The results given in Table I are averages of those obtained with several fibers.

Under optimum conditions, which are not defined by the present data, there is a striking reduction in the crimp of the air-dry fibers. If Mercer's views [2] on the structure of the orthocortex and the paracortex are correct, the removal of crimp may be due to disorientation of the orthocortex, following disulfide bond breakdown, but the fact that there is an optimum concentration of potassium dichromate at some (possibly all) pH values lends some support to the original hypothesis of preferential attack on strained disulfide bonds.

TABLE I

Normality of acid	Concentration of potassium dichromate (%)	Reduction in crimp (%)
0.4	0.2	27
	1.0	57
	5.0	17
4.0	0.2	3
	1.0	24
	5.0	60
8.0	0.2	23
	1.0	42
	5.0	24*

* Obtained after treatment for 30 min.; the fiber was destroyed in 1 hr.

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J. P. E. HUMAN
J. B. SPEAKMAN

Note on the Construction of Fiber Hygrometers

ONTARIO RESEARCH FOUNDATION
Textile Department
Toronto, Canada
September 4, 1953

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

The sensitive element in a fiber hygrometer usually consists of a strand of hair which changes in length with humidity and may be used to operate a needle or pen which registers these changes. The important factor, therefore, in such a hygrometer is the way in which the length of a hair varies with relative humidity. An investigation shows that this is far from ideal for this application.

The best figures for the longitudinal swelling of hair are those given by Stam, Kratz, and White [2], illustrated in Figure 1. Unfortunately, this work was not done on a stabilized fiber, and on desorption the length did not return quite to its original length. This would tend to exaggerate somewhat the hysteresis between absorption and desorption. These curves have two bad features: first, the large hysteresis; and second, the fact that the slope decreases markedly at high humidities, so that about 80% of the total swelling takes place in the first 60% of relative humidity. The hysteresis results in a lack of reliability when the hygrometer is exposed to widely fluctuating relative humidities such as are encountered in a normal room or outside. The flattening results in a scale which is crowded at high humidities and expanded at low humidities.

Ideally, the characteristics most suitable for a hygrometer would probably be a linear relationship between longitudinal swelling and relative humidity and no hysteresis. Such a relationship does not exist for any fiber so far as is now known, but it is possible to approach this ideal with normal nylon 66. The longitudinal-swelling curve for nylon is shown in Figure 2 [1]. It is slightly concave upwards, and shows no appreciable hysteresis. The magnitude of the swelling is about 50% greater than that for human hair.

A recording hygrometer in our laboratory has been fitted out with a single 15-denier nylon filament, and its behavior seems to be completely satisfactory. Hair hygrometers have acquired the reputation of being unreliable, slow in responding to humidity changes, and in general only the crudest of humidity-measuring instruments. There seems to be no reason why a reasonably satisfactory fiber hygrometer could not be made using nylon as the humidity-sensitive element in place of hair. The only precaution necessary would seem to be to relax the nylon thoroughly before mounting, by a heat treatment such as immersion in boiling water for 15 minutes.

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NORMAN J. ABBOTT

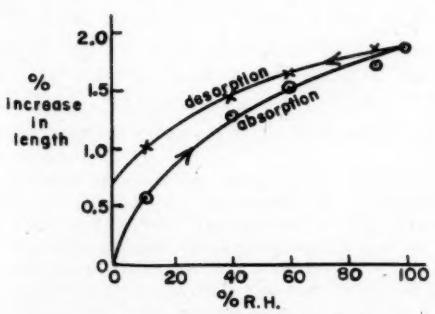

FIG. 1. *Human hair.*

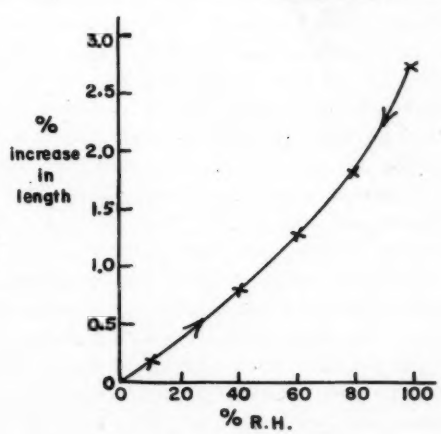

FIG. 2. *Nylon.*

Drape-Flex Stiffness Tester

FABRIC DEVELOPMENT TESTS
185 26th Street
Brooklyn, N. Y.
November 2, 1953

To the Editor
TEXTILE RESEARCH JOURNAL

Dear Sir:

The excellent article in the October, 1953, TEXTILE RESEARCH JOURNAL, entitled "A Comparative Study of the Tinius Olsen and Peirce Stiffness Testers," by Walter J. Hynek and Gerald Winston, of the Quartermaster Research and Development Laboratories, described a new cantilever stiffness tester based on the original mathematical formulas developed by F. T. Peirce. This new, commercially available tester, developed for the U. S. Navy Clothing Supply Office and described in Federal Specification CCC-T-191B, is now properly known as the "Drape-Flex Stiffness Tester" (patent pending) and is available from Fabric Development Tests.

The comparative study by Hynek and Winston concluded that, in comparison with the Tinius Olsen tester, the new cantilever tester was as sensitive and accurate, much simpler, easier to operate, and six times as fast, *but* limited in its ability to measure the wide range of stiffnesses of military fabrics. I wish to point out that the new tester has a much greater range than the indicated 4.2 to 14.6 in.-lbs.$\times 10^{-4}$. The 14.6 value was obtained for a heavy, 11.7-oz./sq.yd. cotton fabric. This value corresponds to a fabric having a projected bending length of only 2.75 in. With the standard 6 in.$\times$1 in. test strip, a projected length of almost 6 in. can be realized. For a fabric weighing 11.7 oz./sq.yd., a 6-in. projection would represent a flexural rigidity of about 150 in.-lbs.$\times 10^{-4}$. Any very unusual material which is even stiffer than this can be measured by using an 8 in.$\times$ 1 in. test strip. The only change required is the addition of 2 in. to the obtained scale reading. This allows the measurement of stiffness values up to 250 in.-lbs.$\times 10^{-4}$. Using the Hynek-Winston conversion factor, this would correspond to almost 800 in.-lbs. $\times 10^{-4}$ on the Tinius Olsen tester. The stiffest apparel fabric I know of that has been tested on the Drape-Flex Stiffness tester was a 20 oz./sq.yd. kersey. This gave warp values of only 30 to 35 in.-lbs.$\times 10^{-4}$. Sheet plastic has been measured up to 88 in.-lbs.$\times 10^{-4}$.

On the soft side, unsized marquisette and 0.00025-in. plastic film have been measured, and gave values of less than 0.1 in.-lbs.$\times 10^{-4}$. With materials in this range, it has been found desirable to reverse the normal testing procedure by starting out with a projection exceeding the bending length of the sample. The speciman clamp is then moved back towards the zero point until the free end of the sample comes *up* to the 43° slope. Generally, a starting projection of 2 in. is sufficient.

J. J. PRESS

INDUSTRIAL SECTION

Improvement of Luster of Cotton

Part III: Correlation of Visual and Physical Aspects of Luster*

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Harris Research Laboratories, Washington, D. C.

Abstract

Luster is not an "absolute" property of the cotton or the yarn, but depends on the sheerness of the fabric, as well as on the weave pattern. One of the reasons for the greater apparent luster of sheer fabrics or thin layers of yarn is that there is less opportunity for light to be scattered within thin layers than in its return from the lower parts of thick layers of yarn. Such scattering adds an approximately equal amount to the reflectances at all angles and so lowers the apparent contrast ratio for the thicker layers.

At the opposite extreme, the reflectance of individual yarns can be determined, and it is demonstrated that yarn removed from the fabric has nearly the same luster as the original yarn, if the weaving crimp is removed. Weaving crimp by itself reduces the measured luster, tending in the same direction as the effect of cross-yarns.

The measurement of contrast ratio in yarns correlates with the average of numerous judgments by observers. For fabrics, the ability of the eye to see sparkle or high lights, and to find the angle of maximum reflection, gives visual judgment some features not possessed by the more simple types of physical measurement, although even with fabrics there is a large degree of correlation.

LUSTER is one of the features of premium-quality cotton fabrics, and also one which makes an appeal at the point of sale. Since the sales appeal of luster is purely through subjective judgment, it is important to examine the relation between physical measurements of optical properties and subjective judgments made on the same range of fabrics.

The basic visual or subjective judgment is judgment of brightness in varying directions, and, similarly, the basic physical measurement is directional reflectance. Since the appearance of high lights, or the recognition of a tendency to concentrate more light in certain directions, like a mirror, and less in others, is a large part of the idea of luster, the physical measurements are also combined in a way which indicates contrast. The contrast ratio is used as a physical index of luster, and is defined as the reflectance at 45°, with illumination at 45°, divided by the reflectance at 45°, with illumination at right angles to the fabric surface. In other words, contrast ratio is a measure of the tendency to reflect like a mirror, in comparison with the scattering of light at other angles. However, it has been pointed out [1, 3] that other properties, such as general level of brightness and transparency or sensation of depth, are involved in the subjective impression. The present paper reports on measurements and comparisons with visual judgment which indicate that transparency, and scattering from the deep layers of the material, are indeed important and have measurable effects on luster.

* A report of work done under contract with the U. S. Dept. of Agriculture and authorized by the Research and Marketing Act of 1946. The contract is being supervised by the Southern Regional Research Laboratory of the Bureau of Agricultural and Industrial Chemistry.

Parts I and II appeared in the July, 1951, and January, 1953, issues of TEXTILE RESEARCH JOURNAL, respectively. Part IV appears in this issue (p. 67).

Relations between Reflection, Absorption, and Scattering

Mention was made in Part I [2] of the need to have a number of layers of yarn to eliminate effects of background. Further study has shown that the effects involved are more complex than had been realized at first, and raise some basic questions regarding correlation of physical measurements and visual appraisal. The chief questions arise from the effect of light which penetrates into underlying layers, and is there reflected, scattered, or absorbed. Owing to the curved path of fibers in the yarns, as well as to the curvatures and irregularities of the reflecting surfaces of the individual fibers, it can be expected that the light reflected back from the lower layers will become more and more scattered as to direction the greater the depth of penetration and return.

To examine this effect, yarn was wound on open frames, so that the only background effect was that of similar layers of yarn, separated by the thickness of the frame, which was 0.055 in. Figure 1 shows the effect on the directional reflectances at the standard combinations of angles, for a natural or "grey" yarn and the same yarn mercerized and gassed. Figure 2 shows the effect on contrast ratio, as the number of layers or cover factor increases.

Figure 2 also shows the effect of inserting a black or a white card in the central space between the layers of mercerized yarn (which, of course, reduces the cover factor to half its value for the open frame). The curve for the black background is very close to that for the open frame. By the time the cover factor has reached 100, the contrast ratio for the white background has converged on the curve for the open frame. Similar results (not shown) are obtained for the natural yarn.

Inspection of the rather parallel course of the two reflectances, in Figure 1, as the number of layers increases, suggests that the difference between the two contains two components, one of which arises from the higher mirrorlike reflectance at 45°, and does not change with increasing number of layers, and the other being a diffuse component which does change with added layers, but to the same extent at both sets of angles. The increase in diffuse light can be thought of as arising from light which penetrates into the cotton, is reflected and refracted within the mass, and emerges scattered in all directions. This diffuse component levels out at the same degree of cover for each set of angles, this being the depth of cotton which absorbs so much of the light, on the journey in and out again, that no more light is returned from greater depths.

The effect of this diffuse component on the contrast ratio is to decrease it, as seen in Figure 2. This follows from the fact that adding an equal amount (the diffuse component from added layers) to both numerator and denominator of any fraction will bring it closer to unity.

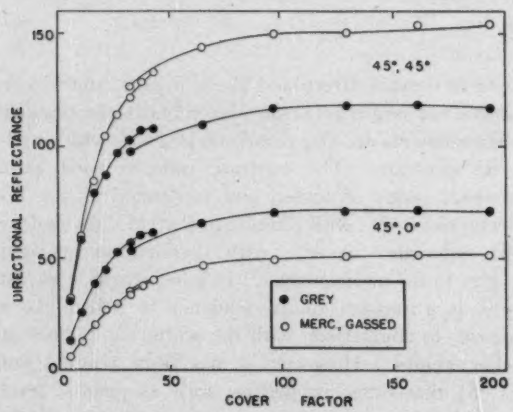

FIG. 1. *Effect of amount of yarn or thickness of specimen on measured reflectances in two directions. The first, closely spaced groups of points were obtained by winding yarn at 12 yarns/inch, in successive layers. The wider spacings were another series, at 72 yarns per inch, also on open frames. Grey cotton—that is, natural Acala 4-42—compared with the same yarn mercerized and gassed; commercial yarn.*

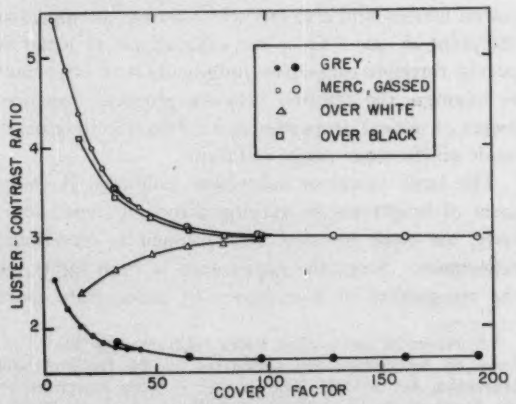

FIG. 2. *Effect of amount of yarn, and of background, on contrast ratio. The results over white or black cards are shown for the mercerized yarn, in addition to those on the open frame.*

These observations indicate that the practice of examining yarn in sufficient depth to rule out background effects overlooks one of the variables encountered in judging cloth made out of the yarn. It can be noted that the more lustrous fabrics tend to be the more sheer ones. Also, if thin windings of yarn and thicker ones are examined side by side, the thin winding will be judged more lustrous, corresponding to the indication of the contrast ratio.

This might indicate that more attention should be given to the rate of change of reflectance or contrast ratio with change of cover or amount of yarn examined. This may well be, but trials of comparing grey and mercerized yarn, or grey and bleached yarn, indicate that the rate of change is much the same for all kinds of cotton yarns, and that any differences between cottons or cottons which have had different finishing procedures lie close to the limits of measurement with the present equipment.

Measurement on Individual Yarns

The extreme to which the reduction of cover factor can be carried is, of course, the examination of individual yarns, or, more logically, even fibers, although this last step has not been made in this work. It has proven possible to examine individual yarns, although the equipment used imposed one experimental difficulty, in that the intensity of the light beam was not uniform over the whole illuminated area. This nonuniformity makes no difference as long as similar whole areas are being examined, but requires additional measurements to enable it to be averaged out as a random variable in comparisons of different types of yarns as individual strands.*

* An instrument described by Olson, Hume, and Reese, of DuPont Experimental Station [4], measures light reflected from individual yarns in high-count fabrics such as rayon-lining twills, and indicates that an instrument could be designed specifically for individual yarn measurements.

The individual yarn method has been used to compare the properties of yarn removed from fabric with that of the original yarn, using the materials of Figures 3 and 4 of Part I [2]. This group of fabrics has been restudied, using yarns removed from the filling. As removed from the fabric, these yarns carry with them a large part of the weaving crimp. For this reason, in one series of tests the yarns were mounted in the holders with only sufficient tension to hold them in a straight average line, but without attempting to remove the weaving crimp by tension. In additional measurements the crimp was removed from the same section of yarn by wetting-out in water and drying in air under slight tension. The results of both types of measurements are shown in Table I.

The results show that the contrast ratio of the yarns removed from the fabric is considerably increased by removing the crimp. For comparison, similar measurements were made on individual yarns taken from the tubes of yarn corresponding to each fabric. These are very close to the measurements made with crimp removed.

Since the effect of crimp in the original fabric appears significant, the possibility of an influence of slight variations of the positions of the individual yarns away from the orientation in the fabric was investigated. For the fabric made from yarn mercerized at natural length, the measurement with the yarns as they appear in the fabric was supplemented by one in which the same yarns were turned around on the yarn axis. With this rotation, of course, the yarn is being viewed in the direction in which it cannot normally be seen—namely, sidewise from within the fabric. This arrangement appears to give an increase in contrast ratio which, however, is smaller than the difference produced by complete removal of the crimp. Hence, it is concluded that

TABLE I. COMPARISON OF INDIVIDUAL YARNS, YARN WINDINGS, AND FABRICS

Type of measurement	Notes	Contrast ratios		
		Grey	Mercerized (natural length)	Mercerized (4%–5% stretch)
Individual yarn*	From fabric (filling):			
	(*a*) as viewed in fabric	2.62	3.39	5.09
	(*b*) turned 90° on own axis	—	3.77	—
	(*c*) crimp removed	3.03	4.90	6.43
	From tube	2.96	4.81	5.78
Windings on cards		1.72	2.26	2.67
Fabric	Parallel filling (48 per in.)	1.24	1.33	1.42
	Parallel warp (44 per in.)	1.15	1.16	1.19

* Average of 10 sets of measurements.

the proper procedure for viewing yarns removed from fabrics is to remove the crimp.

For comparison, the data obtained earlier by winding these yarns onto cards has been added to Table I. It will be noted that the contrast ratios on cards and as individual yarns for the two mercerized yarns are very nearly proportional and that the contrast ratios for the grey yarn are nearly in the same proportion. In other words, the individual yarn measurements run in the same order, but higher, than the contrast ratios determined from windings. The fabric data are also shown, to indicate how much more clear-cut the differentiation of luster is in yarn than in fabric.

The most important point regarding this test is that it demonstrates the applicability of a powerful tool by which one can compare visual ratings of fabric with the properties of the yarns. These results confirm the earlier conclusion that differences of luster can be measured better, as far as physical measurements go, on the yarn rather than on the fabric.

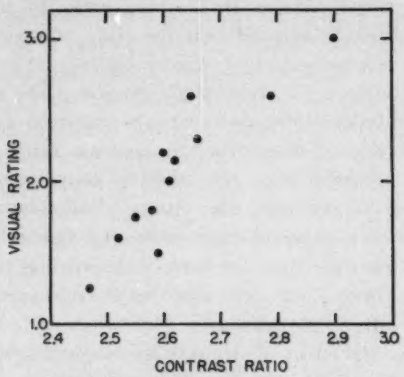

FIG. 3. *Correlation of visual rating of luster, and contrast ratio, in yarns wound on flat cards. This is a series of yarns, all one lot of Deltapine cotton, mercerized at a series of lengths from 97% to 104% of original length.*

Correlations between Visual Judgment and Physical Measurement in Yarns

A number of comparisons have been made to test the relationship between judgments of luster and the results of measurement. The windings of yarn used for measurement were submitted to observers who were asked to sort them into groups, in order of luster. The observers varied in their experience in judging luster, from workers on the problem who had been accustomed to handling the yarns and making judgments, to others whose background for this purpose could be regarded as that of typical ultimate consumers. In the trials with yarns, three groupings were adequate—low, middle, and high, with scores of 1, 2, and 3, respectively. The average score for each winding of yarn, from the rankings given by a number of observers, permits a more finely scaled

TABLE II. STRUCTURE AND LUSTER OF FABRICS

Fabric	Weight (oz./yd.²)	Threads/inch Warp	Threads/inch Filling	Visual rank	Contrast ratio 1 layer	Contrast ratio 8 layers
Comparisons, plain weave						
Silk	1.4	104	84	5	2.00	1.69
Linen	1.9	99	84	5	1.73	1.40
Plain-weave, flat cotton						
Batiste	1.5	112	88	5	2.19	1.60
Batiste	1.5	112	100	5	1.59	1.31
Batiste	2.0	96	96	3.6	1.57	1.24
Batiste	1.9	100	96	3.4	1.52	1.23
Nainsook, merc.	2.1	100	96	3.2	1.51	1.30
Fine lawn	2.0	100	76	3	1.26	1.15
Print cloth	3.7	88	84	2.6	1.36	1.19
Balloon cloth	3.8	128	108	2.6	1.34	1.21
Longcloth	3.3	84	78	2.4	1.52	1.37
Ribbed cotton						
Poplin	4.5	108	50	4.0	1.19	1.12
Twills						
Gabardine, 2/1	3.5	108	64	2.6	1.34	1.21
Gabardine, 3/1	6.3	108	60	2.6	1.14	1.12
Gabardine, 3/1	7.2	88	42	1	1.14	1.10
Gabardine, 2/1	6.9	80	48	1	1.22	1.19

ranking than one observer can give in one trial. A typical correlation diagram is shown in Figure 3. This represents the average of five judgments by one observer on a series of yarns mercerized with increasing degrees of stretch.* The results in this series indicate that differences of contrast ratio as small as 0.04 can be recognized by comparing windings on separate cards. However, even smaller differences can be recognized when yarns of one kind are woven or knit adjacent to another kind.

In one example, tubing knit of alternating strips of Deltapine 14 and Acala 4–42 yarns,† which had contrast ratios of 1.67 and 1.68, respectively, showed one strip brighter under one condition of view, and the other under another, indicating that even this small luster difference could be observed. In general, in woven or knit goods, if change of direction of illumination reverses the order of rank in light and dark between parallel sets of yarns, it indicates that one set has a higher peak of reflectance near the mirror angle, and less scattering at other angles, than the other. This, of course, gives rise both to the differences in luster as observed and to a difference in contrast ratios.

* Deltapine 14 cotton, mercerized at the Southern Regional Research Laboratory at intervals of 0.5% stretch, from 97% through 102% of original length, with an additional specimen at 104% of original length. These yarns were made available by Mr. C. F. Goldthwait.

† Material made available by H. A. Rutherford and K. S. Campbell of North Carolina State College School of Textiles.

Correlation in Fabrics

To give a general view of the problems of correlation of visual judgments of luster with physical measurements in fabrics, a group of white cotton fabrics were purchased at retail. These ranged from sheer batiste at 1.5 oz./yd.^{2}, to work-clothing twill at 7 oz./yd.^{2}, in three groups of weaves: (*1*) flat plain weave, (*2*) strongly ribbed plain weave, with heavy fillings and fine warps (poplin), and (*3*) twills. In addition, a light silk and a fine linen plain-weave fabric were included for comparison. Structural data for these fabrics are given in Table II.

These fabrics were also judged visually by five observers, who were asked to group them in five groups, from low through medium to high luster, with two intermediate groupings. These groupings were given scores from 1 for low to 5 for high, and the average for each fabric was determined. The fabrics were submitted in folded form, eight or more layers thick, but it is believed that most observers judged the effect of the top layer as an individual layer, rather than the effect of the background mass, since the correlation with the measured results on one layer appears stronger than that with eight layers.

Figure 4 shows the relation between contrast ratio for one layer of fabric and the visual rating for all the plain-weave flat fabrics and the twills, plus one of the ribbed poplins. There is a general trend of correlation to which the poplin is an exception. This

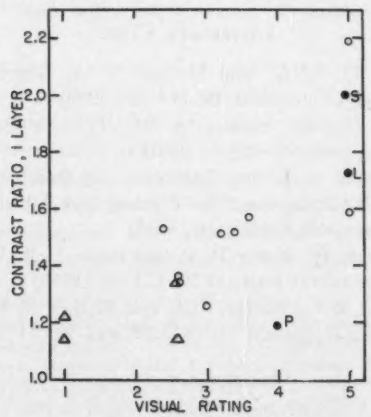

FIG. 4. *Correlation of contrast ratio and visual rating of luster. The fabrics are all white cotton fabrics, except* S, *a thin silk, and* L, *a linen. The open circles indicate plain-weave, flat fabrics; the triangles, twills. The solid circle marked* P *is a very lustrous poplin, strongly ribbed, and is an exception to the general trend.*

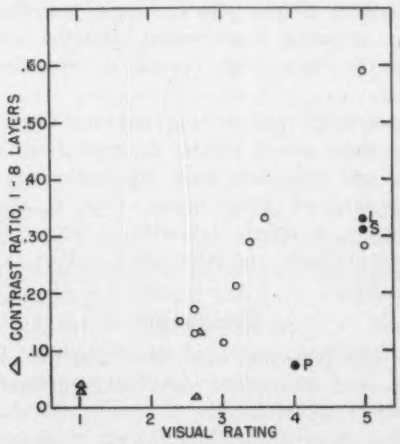

FIG. 5. *Correlation of sheerness and visual rating of luster. Sheerness is indicated by the difference between contrast ratio of 1 layer and 8 layers of fabric. These are the same fabrics as in Figure 4, but here the correlation is somewhat stronger.*

particular poplin is judged high in luster, because the sparkle and high light of the individual yarns can be seen by the eye, although the type of instrument which obtains an average over an area which is large with respect to the weaving unit fails to recognize this contrast.

Figure 5 shows the difference in contrast ratio, on going from one layer to eight layers, plotted against the same visual ratings. This decrease in contrast ratio is a measure of the transparency or sheerness of the fabrics, and shows the same general trend and pattern as does the contrast ratio itself. From a strictly logical point of view, this does not demonstrate that sheerness is involved in the judgment of luster, because it could be regarded as a chance or bias of this particular group of fabrics. However, this group is believed to be representative of a general trend in fabrics. The value of transparency is also recognized practically in the trade, in such terms as "soft" or "deep-seated" luster.

There is another respect in which observation may differ from physical measurement, in that the eye includes the absolute peak of reflectance, in visual observation, while measurements at the mirror angle are not peak measurements for cotton fabrics, as pointed out by Quynn, Bernet, and Fischer [5]. However, when the peak is located by the instrument, it is always found to be farther from the mirror angle, and relatively less high with respect to the mirror-angle reflectance the lower the luster. The location of the peak is not a convenient or sensitive physical measurement, and the measurement at the mirror angle appears to serve the same purpose.

More detailed studies of the relation of fabric structure to luster would require determination of yarn number and twist, and more information regarding the finishing of these fabrics than is available. Fortunately, a series of fabrics of known history has been available, and is reported in Part IV (this issue, p. 67).

Conclusion

The data presented here show that the physical measurement of contrast ratio is, in general, correlated with visual judgment of luster, for white cotton yarns and fabrics. One important qualification is that sheerness or transparency is also a contributing factor, affecting both the contrast ratio and the visual judgment in the same general way. Another important qualification is that the complications of fabric structure must be considered. This is especially true of fabrics which are strongly ribbed, as in poplins, where the eye can see the sparkle and high light of the yarns, but the effect of the cross-yarns may dominate the measurements. In general, measurements on yarns are more significant with regard to luster than measurements on fabrics, and in critical cases it would be desirable to ravel yarns out of the fabric, or to use a fine scaled system of measurement, in which the area examined is reduced to one yarn by itself as it shows in the unit of weaving pattern.

However, for many purposes of comparison, measurements on fabrics themselves by the methods used here can be useful.

Acknowledgment

We wish to express our thanks to Messrs. J. D. Dean, C. F. Goldthwait, and E. M. Buras, Jr., of the Southern Regional Research Laboratory, who have aided this work by their guidance and advice and have made available experimental material.

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Improvement of Luster of Cotton

Part IV: Effect of Weaving Pattern on Luster in a Series of Bleached Cotton Fabrics*

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Abstract

One of the means of increasing luster, which is available to designers working in any fiber, is variation of weaving pattern or construction. It is examined here for comparison with other means of improvement which are more specifically applicable to cotton. The increase in luster in going from plain weave to a 4/1 float is considerable; a 4/1 float is significantly higher in luster than a 3/1 float, all at constant texture, with the same yarns.

Sheerness obtained either by finer yarns or decreased texture also increases luster. However, if cover is kept constant by using more of the finer yarns, the two tendencies oppose each other, reducing the change of luster.

Introduction

Luster is one of the factors in the selling of fabrics which can be influenced by fabric design. There are some signs of a style trend or fashion interest in "white on white" in men's shirtings, in which the pattern effects are secured mainly by contrast of plain-weave areas with areas of other weaving patterns, in which the float length—that is, the number of yarns crossed over between interlacings—is increased. In table linen and upholstery fabrics, it is also usual to secure contrast not only by variation of float length, but also by contrast of warp and filling floats.

While the "white-on-white" shirtings and damask patterns in tablecloths and upholstery present to the eye the contrast of relatively small areas of pattern, it simplifies measurement to have larger, uniform areas of fabric representing the different patterns. Such a group of fabrics had been prepared by the Southern Regional Research Laboratory, and were made available for examination for luster.† This group of fabrics varies in weaving pattern from plain weave to 4/1 twill, and also varies in number of ends and picks and in yarn fineness. All are from the same lot of cotton, and have been processed similarly, but have not been mercerized.

In the subgroup designated as "phase *I*," this group of fabrics shows variations in weave pattern, other factors being constant. In phases *II* and *III*, the tightness of weaving was changed, either by changing the texture—that is, the number of yarns/inch—or by changing the size of the yarns, or both. The combined effect of changes of texture and yarn size can be expressed in the cover factor, defined as number of yarns/inch divided by the square root of the cotton yarn number [2], which is related to the degree to which the yarns, lying side by side as spaced in the fabric, would cover a surface. In phase *II*, for Oxford and twill weaves, the texture was reduced, using the same yarns. For the sateen, however, the number of picks was substantially increased, with a small reduction in warp ends/inch. In phase *III*, for Oxford and twill, coarser yarns were used, but at the same texture as in phase *II*, so that the effect of changing cover factor by changing yarn diameter alone could be examined.

In addition, since the corresponding yarns were made available, the effect of the crosswise system of yarns always present in fabric plus the effect of weaving crimp or undulation of yarns crossing each other could be compared with measurements on yarns by themselves.

* A report of work done under contract with the U. S. Dept. of Agriculture and authorized by the Research and Marketing Act of 1946. The contract is being supervised by the Southern Regional Research Laboratory of the Bureau of Agricultural and Engineering Chemistry.

Parts I and II appeared in the July, 1951, and January, 1953, issues of TEXTILE RESEARCH JOURNAL, respectively. Part III appears in this issue (p. 61).

† These fabrics and yarns were made available by Mr. R. J. Cheatham of the Southern Regional Research Laboratory, U.S.D.A. The fabrics had been prepared by the S.R.R.L. for a cooperative study with the Bureau of Home Economics and Human Nutrition.

TABLE I. EFFECT OF WEAVING PATTERN IN BLEACHED FABRICS *

Fabric type	Warp floats over	Luster (contrast ratio) Parallel to warp 1 layer	Parallel to warp 4 layers	Parallel to filling 1 layer	Parallel to filling 4 layers
Broadcloth	1	1.44	1.28	1.33	1.18
Oxford (2 ends over 1 pick)	1	1.40	1.27	1.34	1.20
Twill 3/1	3	1.55	1.36	1.32	1.23
Sateen, 5 harness warp	4	1.62	1.42	1.40	1.18

* All were woven 128×68, 40's warp, 42's filling (phase *I*). All were measured on warp float face.

The fabrics were woven 14 in. wide in the grey, and bleached and finished at 13.75 in.

Methods

The luster is measured in terms of the relative amount of light reflected in different directions, by the "contrast ratio," which is

$$\frac{\text{reflectance for illumination at } 45°, \text{ viewing at } 45°}{\text{reflectance for illumination at } 0°, \text{ viewing at } 45°}$$

where 0° is the line "normal" or perpendicular to the surface. Thus, the contrast ratio is the reflectance near the peak or high light, as represented by the angle combination for mirrorlike reflection, divided by the reflectance at a direction representing general scattering tendency. The higher the contrast ratio, the more mirrorlike is the reflecting tendency of the surface, and the higher is the luster. The optical methods have been described elsewhere [1].

In the reflectance measurements, the fabrics were all examined on the face having warp floats, with the plane of the path of the light parallel to the warp in one set of measurements, and to the filling in another. The specimens were mounted across an opening in a frame, so that any effect of backing by other materials would be absent.

In visual rating, the fabrics were also examined on the face, and primarily in the warp direction, although observers would vary the angle of view from parallel to the warp through a variety of angles as they made their comparisons. The fabrics were folded so the effect of backing by numerous layers was being judged, at least in part. The observers were asked to rank the fabrics within groups, and the rankings were given scores, from 1 for least luster, upwards for higher luster, the highest score being the same as the number of fabrics compared.

TABLE II. EFFECT OF INCREASING NUMBER OF LAYERS

Fabric	View parallel	Layers	Reflectance* 45°, 0°	Reflectance* 45°, 45°	Contrast ratio
Broadcloth (128×68, bleached)	Warp	1	59.5	86.0	1.44
		2	72.5	96.5	1.31
		3	78.0	101.0	1.30
		4	78.5	100.5	1.28
	Filling	1	61.0	82.0	1.33
		2	77.5	95.5	1.28
		3	80.5	96.5	1.21
		4	81.0	97.5	1.18
Sateen (128×68, bleached)	Warp	1	59.0	95.5	1.62
		2	72.0	107.5	1.48
		3	78.0	112.5	1.45
		4	80.5	114.5	1.42
	Filling	1	65.5	84.5	1.40
		2	76.5	96.0	1.23
		3	84.0	101.5	1.20
		4	86.5	102.0	1.18

* On scale MgO surface = 100. This is a nearly ideal matt white.

Results

Effect of Weave Pattern

The effect of weaving pattern, other factors being constant, is shown in Table I. The dominant influence is the length of float parallel to the direction of view. Thus, for viewing parallel to the warp, there is a large gain in luster in going from the float length of 1, for plain-weave broadcloth or for Oxford, to the twill or sateen. Floating over 4 picks, in the sateen, gives more luster than over 3, in the twill.

The relations along the filling are but little changed by variation in the warp floats. This is especially the case for four layers of fabric. However, one of the measurements, that for one layer of sateen, in the "parallel to filling" set is higher than all the others. The reason for this somewhat higher value for sateen examined in a single layer is not at once apparent. This increase is considerably less than

TABLE III. EFFECT OF CHANGES IN TEXTURE (ENDS×PICKS) OR IN YARN SIZE

Fabric type	Phase	Texture (as woven)	Yarn No. Warp	Yarn No. Filling	Luster (contrast ratio) Parallel to warp 1 layer	Parallel to warp 4 layers	Parallel to filling 1 layer	Parallel to filling 4 layers	Cover factor* Warp	Cover factor* Filling
Oxford (2 ends over 1 pick)	*I*	128×68	40	42	1.40	1.27	1.34	1.20	20.2	10.8
	II	88×42	40	42	1.60	1.29	1.41	1.22	13.9	6.5
	III	88×42	40	15	1.36	1.21	1.36	1.20	13.9	10.8
Twill 3/1	*I*	128×68	40	42	1.55	1.36	1.32	1.23	20.2	10.8
	II	100×60	40	42	1.60	1.38	1.33	1.17	15.8	9.3
	III	100×60	31	32	1.56	1.36	1.30	1.18	17.9	10.6
Sateen 4/1	*I*	128×68	40	42	1.62	1.42	1.40	1.18	20.2	10.8
	II	120×84	40	42	1.58	1.39	1.28	1.17	19.0	13.0

* Cover factor = yarns per inch/square root of yarn number. A cover factor of 28 would represent yarn touching yarn, with 100% of the available area covered by projected yarn diameters, for cotton yarns of normal density, according to Peirce [2], if the yarns were uniform and circular (which they are not).

the increase in contrast ratio in the warp direction accompanying longer warp floats. Visual judgment confirms the measurement, that this fabric indeed is more lustrous in the filling direction. A possible explanation is that the sateen appears to be a flatter fabric, in which the filling may lie straighter than in the twill, since in the sateen the float length for both sets of yarn is longer, and the angular deformation of each yarn at the crossover points can therefore be less.

Effect of Number of Fabric Layers

These fabrics are relatively thin and open, so that considerable light passes through one layer of fabric. In order to make comparisons of the fabric under conditions similar to those used for yarns, in which the effect of variation in amount of material is eliminated as far as possible by using numerous layers, measurements were made with increasing number of layers of fabric. Table II shows representative results for the reflectances in the individual directions, as well as the contrast ratios, as the number of layers of fabric is increased. The effect of additional material is generally completed at four layers of thickness.

The chief effect of additional layers is to increase the reflectance in both combinations of angles. With the second and third layers, the increase in the general scattering (45°, 0°) is often slightly greater than the increase in the mirror direction (45°, 45°); with the fourth layer, the change in each is small and approaches the level of variation in sampling and measurement. Adding the same amount to numerator and denominator of any fraction will bring it closer to unity, which accounts for part of the trend of contrast ratio. In addition, the denominator, representing general scattering, increases somewhat faster at first, which adds to the downward trend of contrast ratio.

TABLE IV. EFFECT OF TEXTURE ON REFLECTANCE

Fabric	Texture (as woven)	Layers	Reflectances parallel to warp 45°, 0°	Reflectances parallel to warp 45°, 45°
Oxford, *I*	128×68	1	60.5	85.2
II	88×42	1	42.2	67.7
I	128×68	4	81.2	103.5
II	88×42	4	75.7	98.0
3/1 Twill, *I*	128×68	1	61.7	96.0
II	100×60	1	52.5	84.0
I	128×68	4	81.5	110.7
II	100×60	4	77.5	107.7

Comparison of Effects of Texture

The effect of variation in number of yarns/inch, the fineness of the yarns being constant, is shown by comparison of the phase *I* and phase *II* groups in Table III. In the Oxford weave, reducing both ends and picks results in a higher contrast ratio, for either one or four layers of fabric. However, part of this arises from an effect similar to that of changing the number of layers of an individual fabric. The low-texture Oxford of phase *II* is lower in absolute amount of light being reflected at both one and four layers, as shown in Table IV.

Visual examination of these two Oxfords shows that the phase *II* is very open and thready, almost gauzelike, while the phase *I* Oxford has the appearance of a fabric with a normal cover or solidity. The

individual threads are hardly noticeable in the higher texture. Visual judgment of luster alone, as distinguished from total amount of light reflected, confirms the measurement, and may indicate that the threads run straighter at the low texture, and also that there is less general scattering to distract the eye.

In the 3/1 twill weave, the effect of reducing the texture (phase *II* compared with phase *I*) is also to increase the contrast ratio, in the warp direction, for single layers of fabric, but there is almost no increase for four layers together. One reason for this is shown in Table IV, which indicates that the individual reflectances change at different rates for twills than for Oxfords, and have come nearer to each other at four layers for the twills. The cover factors shown in Table II indicate that the lower-texture twill is a more solid fabric than the low-texture Oxford, and visual inspection, while indicating that both low-texture fabrics are sleazy, confirms that the twill has relatively better cover. Hence, another way of describing the reason for less change in contrast ratio with change in texture for the twill is that the difference in texture is less.

Increase of texture is shown in the 4/1 sateen series, for the filling direction, with only a slight reduction in the warp. There is little change in warp contrast ratio, but the usual effect of decreased texture is reversed. This is probably caused by increased general scattering from the larger number of crosswise (filling) yarns. The filling shows the usual effect; the higher the cover, the lower the contrast ratio.

Effect of Yarn Fineness or Coarseness

The effect of coarse filling yarns is shown by phase *III* of the Oxford weave. The increased scattering from these filling yarns, plus the greater bending of the warp yarns, reduces the warp-direction contrast ratio. Visual inspection in the warp direction shows that the coarse filling is the most conspicuous feature of the phase *III* Oxford.

In the filling direction, the cover factor is the same as phase *I*, but with coarser yarns. The contrast ratio in the filling direction is also nearly the same, but this is probably the result of the balance of opposing tendencies. The lower warp cover factor in phase *III* gives longer filling floats, and the greater yarn stiffness reduces filling crimp, tendencies making for higher filling-direction luster. However, in general, coarser yarns have lower luster (as shown in Table V), which appears to largely compensate the other tendencies.

In the twill weave, phase *III* keeps texture constant, but increases the size of both sets of yarns. The coarser yarns and higher fabric cover factors show lower luster than the similar texture in phase *II*. Here the effect of increased crimp and decreased free length between yarn intersections may also be decreasing luster, in addition to the effect of yarn coarseness in decreasing luster; at any rate, the general rule, higher cover, lower luster, is seen both warp and filling way for the phase *II* and *III* twills. The effect, if any, is less clear-cut for comparisons with the phase *I* twill, which has nearly the same cover factors as phase *III*.

Comparisons with Yarns

Measurements on the separate yarns are shown in Table V. The contrast ratio for the 40's warp yarn used in phase *I* is 1.38; this is just about the level for four layers of twill or sateen fabric. However, the total cover in the fabrics is less than that in the yarn measurements, so the additional scattering from cross-yarns in the fabric is apparently about equivalent to the additional scattering from deeper layers in the yarn arrangement. The contrast ratio of one

TABLE V. MEASUREMENTS ON YARNS SPUN FROM DELTAPINE COTTON *

Yarn No.	Twist multiplier	Machine twist (t.p.i.)	Use	Total cover factor in specimen	Yarn crossed to view reflectance		Yarn parallel to view Reflectance		Contrast ratio
					45°, 0°	45°, 45°	45°, 0°	45°, 45°	
15's	3.50	13.54	filling	116	70.0	75.5	69.5	97.0	1.39
31's	4.80	26.44	warp	121	71.5	76.5	70.0	96.0	1.37
32's	3.50	19.60	filling	89	73.5	81.0	69.0	100.0	1.45
40's	4.80	29.92	warp	115	70.0	78.5	70.0	92.0	1.38
				20	—	—	34.0	57.0	1.67
42's	3.50	22.74	filling	112	74.0	81.0	70.0	102.0	1.45
				20	—	—	36.0	65.0	1.80

* All measurements were for 7 layers, except for the 40's and 42's, which were also measured in 1 layer on one side of an open frame.

layer of fabric is higher than for seven layers of yarn, but this is much influenced by the difference in total amount of cover. The contrast ratio for one layer of warp yarn, at the same cover as in the fabric, is, however, much higher than one layer of fabric: 1.67 compared with 1.40. This difference indicates the effect of the crosswise yarns and of weaving crimp.

The filling yarns are poorly represented on the warp face of these fabrics, and the scattering from the crosswise warp yarns reduces the contrast ratio of the filling in these fabrics to much below that of even a thick layer of filling yarn.

The reflectances for each set of yarns arranged crosswise—that is, perpendicular to instead of parallel to the plane of the path of the light—are also shown. Both the 45°, 0° and 45°, 45° crosswise reflectances are close to the 45°, 0° for the parallel arrangement. This shows that for 45°, 0° view both sets of yarns should look almost the same in brightness or amount of light reflected, as indeed they do, but that for examination at the mirror-angle condition, the parallel set of yarns will appear brighter.

The measurements on yarns also illustrate the general tendency for luster to decrease with increasing twist, being lower for the two warp yarns. In addition, the luster is lower, at either twist multiplier, for the coarser yarn.

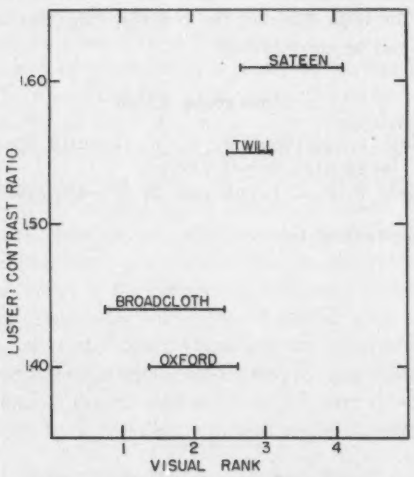

FIG. 1. *Correlation of visual judgments and measurements for fabrics of the same texture and yarns (phase* I*) but differing in weaving pattern, presented as a group. The spread of the bars represents the range of visual rank within which a repeat trial has a 50% chance of falling.*

Too detailed comparison of yarn and fabric at the same cover factor was not rewarding since the yarns were unbleached and the grey fabric had sizing, while the finished fabric may have been calendered, dried on cans, or singed, increasing its smoothness. Also, the contrast ratio of parallel arrays of yarns is rather sensitive to cover factor—that is, to the amount of yarn and number of layers—while measurements on fabrics always involve the crosswise yarns.

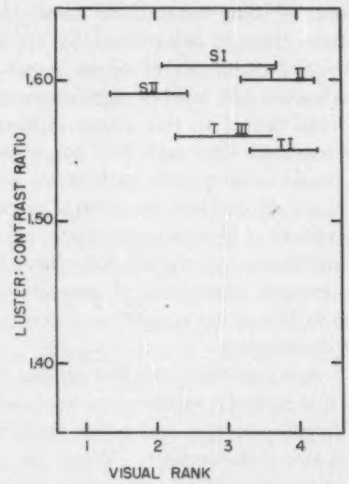

FIG. 2. *Correlation in twills and sateens, presented as a group. Observers and instruments agree in ranking these closely together. Span of the bars indicates 50% probability range.* SI *and* SII *are sateens, and* TI, TII, *and* TIII *are twills, of the "phase" or grouping indicated by the Roman numerals.*

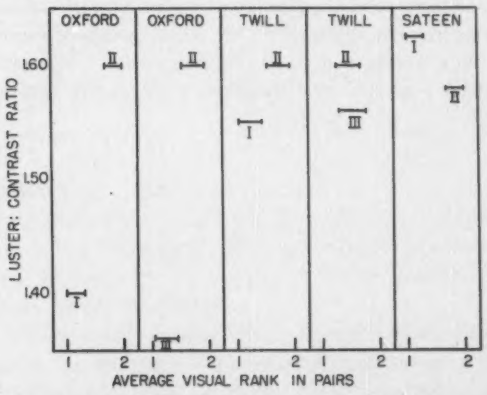

FIG. 3. *Correlation within pairs, each pair presented by itself. The span of the bars represents the 50% probability range. The Roman numerals indicate the "phase" or group for each fabric.*

Correlation with Visual Judgment

The results of visual ranking, averaged for ten observers, are shown in comparison with contrast ratio measured in the warp direction on one layer of fabric in Figures 1, 2, and 3.

Figure 1 shows the four fabrics of phase *I*, all of the same yarns and texture, compared together. The length of the bar extending from each point represents the difference from the observed score, which has a 50% chance of being found if the visual judgments are repeated. This is determined by applying the *t*-test to the individual set of visual scores. Thus, the bars indicate a low level of significance of difference for visual scores in this group of four fabrics considered together, since each 50% bar, extended up or down, would overlap with each of its neighbors, and several would include the central point of the nearer neighbors. However, in spite of the low statistical significance of certain differences in visual score, the general agreement of measurement and observation in this group is good, and in accord with reasonable expectation.

Figure 2 shows all the twills and sateens together, and shows that with the smaller measured differences there are larger overlaps and lower statistical significance of visual differences. Visual judgment is more consistent when fabrics are compared only two at a time, as in Figure 3.

Figure 3 shows, however, that judgments on twills *II* and *III* overlap even when compared by themselves, eliminating the need for considering this reversal of trend with contrast ratio. Twills *I* and *II*, considered by themselves, correspond visually and by measurement, although in the larger group of Figure 2 they overlapped in visual judgments. However, sateen *I* and *II* by themselves confirm the reversal shown in Figure 2, with no overlap. It may be that in this pair the eye is responding to the higher total amount of light reflected with higher total cover factor.

Thus, while there is general correlation of visual judgment and measurement, each type of fabric pair should be checked, as well as the consistency of observers.

Summary

Weaving pattern influences luster through its effects on the length of a given set of yarns lying on the surface, the degree of curvature or straightness of each set, and the number of interruptions by the crosswise set. The longer and straighter the surface floats of the set of yarns which parallels the line of view, the higher the luster.

Decreasing texture and cover, using the same yarns, increases luster. In general, sheer or thready fabrics have higher luster than fabrics made with the same yarns at higher texture or cover.

Coarser yarns decrease luster; finer yarns increase it. Thus, if both texture and yarn size are changed, keeping cover constant, the effect on luster is the result of two opposing tendencies, and the changes are smaller than would result from variation in only one factor.

Visual judgment of luster shows fair correlation with the indications of the contrast ratio, but exceptions can be encountered.

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Yarn Evenness and its Determination*

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Introduction

With spun yarn, evenness is of great practical importance. This has always been the case, as proved by the efforts made in the early days of industrial production to control evenness. A personal estimation of evenness may be obtained by visual control of a yarn showboard. By weighing numerous yarn pieces of equal length, evenness can be measured with physical accuracy; this method has been known for a long time, but it has not often been applied practically because it requires too much work and time.

Of late, evenness measurements have been meeting with increased interest in industry, because modern testing instruments make accurate determination of yarn irregularity easy and because several recent theoretical investigations have disclosed important mathematical laws which throw more light on the subject. Accordingly, physical evenness tests are now more generally used, in agreement with their fundamental importance. The classical method of weighing yarn pieces of prescribed length is still used in special research work, but for practical purposes it is too slow and too costly.

The lack of evenness of a yarn may be due to an irregular number of fibers per yarn cross section or to irregularity of fiber thickness. The irregularity of fiber cross section is obviously different for various fiber qualities. In many cases, however, the fiber cross-section irregularity is known, is rather slight, and is responsible for only a small fraction of the yarn unevenness; unevenness, then, is due mainly to the difference in the number of fibers per yarn cross section. Especially with yarns of cotton, spun rayon, and worsted, calculations based on the assumption of constant fiber thickness, attributing all unevenness to variations of the number of fibers per yarn cross section, may lead to satisfactory and useful conclusions.

Fiber Distribution within Yarns

The number of fibers in a yarn cross section is obviously not constant; its mean value and its law of variability are important characteristics of the yarn structure. With the aid of a microscope, the fiber number can be determined for any given cross section; the easiest way of determining it consists in cutting a short piece of yarn, about .5 mm. long, and counting the fiber pieces, now distinctly separated, with a binocular microscope. This, however, involves the destruction of the yarn.

The yarn structure may also be described by the distribution of the one-sided, say right-hand, fiber ends. The number of right-hand fiber ends per unit yarn length may be determined by actual test or by calculus. By dividing the length unit by the figure thus found, the average displacement from fiber end to fiber end may be obtained. When submitting a sliver to a drafting process, the displacement of the fiber ends will be increased in proportion to the draft.

If the fibers are of uniform length, as is the case with cut staple fibers, the number of fibers per yarn cross section and the number of one-sided fiber ends per unit of yarn length are closely related; if one is known, the other may be derived easily. In dealing with fibers of varying length, an approximate calculation is possible, based on the fiber mean length; however, the accurate calculation, considering the actual fiber length distribution, becomes very complicated and leads only to a probable result, subject to a certain degree of variability.

In the past, the general opinion was that in a perfect yarn the number of fibers per cross section should be constant; accordingly, the spinner's aim was to produce a yarn of absolutely regular fiber distribution. Assuming the yarn to be made of fibers of constant length and thickness, the distribution of the right-hand fiber ends would necessarily also be perfectly balanced—a constant number of fiber ends per length unit.

Mathematical studies [5, 9] of recent years have taught us, however, that even theoretically no such expectation appears justified. Fiber distribution in the yarn follows the laws of probability, which exclude perfect uniformity, even if no error whatever should occur during spinning.

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Some of the fundamentals of the theory shall be given here, without attempting to give a complete derivation. The probability that one of the many millions of fibers going into the works at the same time will cross one particular yarn section is exceedingly small; on the other hand, the probability that the fiber mentioned will not cross this section is very large—*i.e.*, almost equal to unity. Therefore, the fiber distribution within yarn obeys a binomial law, in which the two probabilities are extremely different and the smaller one nearly disappears; this special case of binomial distribution leads to a Poisson series, also known as the "law of small numbers." Accordingly, the number of fibers crossing various yarn cross sections varies along the yarn length, according to a Poisson distribution, if the assumption that the fiber distribution along the yarn is purely random is justified. This assumption is fairly well satisfied by spinning.

The average number of fibers per cross section, n, follows immediately from the yarn count and the mean fiber count, a correction to account for yarn twist being necessary in the case of highly twisted yarns [1]:

$$n = \frac{N_F}{N} \cdot \left(\frac{2 \cos \beta}{1 + \cos \beta} \right),$$

where N_F = individual fiber count, N = yarn count, and β = average inclination of superficial fibers against yarn axis. For ordinary yarn twist, the factor in parentheses is practically equal to unity.

The Poisson distribution is defined by the one constant, n, its mean. Its standard deviation, s, is given by

$$s = \sqrt{n}.$$

Since the number of fibers per cross section obeys the Poisson law, the theoretical value of its standard deviation is known, being equal to the root of the mean.

Very often, instead of the absolute value of the standard deviation, the ratio of the standard deviation to the mean,

$$CV = \frac{s}{n} = \frac{\sqrt{n}}{n} = \frac{1}{\sqrt{n}} = \frac{100}{\sqrt{n}}\%,$$

called the "coefficient of variation," is used. This ratio is a measure of the relative variability.

The standard deviation and the coefficient of variation, as defined above, consider only the irregularities produced by the changing number of fibers per cross section; only if the fiber thickness is constant do they measure the whole yarn irregularity. If the fiber thickness is irregular and its coefficient of variation is equal to V_f, the coefficient of variation of the yarn becomes

$$CV = \frac{100 \sqrt{1 + 0.0001\ V_f^2}}{\sqrt{n}}\%,$$

as shown by Martindale [5] and also by Picard [7]. In reality, the contribution of the fiber thickness variation to yarn irregularity is rather small, as shown by tests on cotton yarns by Foster [3]: $CV = (100/\sqrt{n})\%$, neglecting fiber thickness irregularity; $CV = (106/\sqrt{n})\%$, considering fiber thickness irregularity.

The standard deviation of the probable number of fibers per cross section of yarn mainly depends upon the mean fiber number; the coefficient of variation increases from 1% to 10% and to 20% if the mean fiber number decreases from 10,000 to 100 and to as few as 25. Therefore, in a sliver or roving containing an average of 10,000 fibers per cross section, the irregularity theoretically expected is very small—the probability for cross sections with either less than 9,700 or more than 10,300 fibers is as low as .27%; in a yarn with an average of only 100 fibers per cross section, cross sections will be found with the same probability—having either less than 70 or more than 130 fibers. This is one reason why fine yarns are at particular disadvantage.

The irregularity registered by actual control, however, is higher than the theoretical irregularity; the Poisson standard deviation is to be considered as an ideal limit which could only be realized by a perfect spinning plant. We must consider it impossible to spin a yarn less irregular than that expected according to Poisson's law.

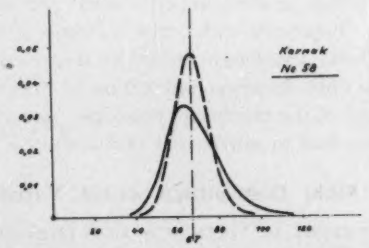

FIG. 1. *Cotton yarn Ne = 58. Fiber frequency distribution in yarn cross section.* Solid curve—*Experimental.* Dotted curve—*Theoretical, calculated according to Poisson law.*

Figures 1 and 2 show the theoretical and the measured fiber frequency distribution for two different yarns. Both yarns were spun from Egyptian Karnak cotton; their main characteristics are given in Table I.

The graphs as well as the table show that the actual standard deviation is higher than the theoretical one; the difference is to be attributed to the inevitable imperfection of the spinning plant, disturbing a completely random distribution of the fibers. In the two yarns considered, the difference between the theoretical and the actual values for the standard deviation is small, and both yarns, accordingly, are of very good quality. The theoretical and the experimental curves of fiber number frequencies show a certain affinity, which tends to prove the correctness of the above remarks. The experimental frequency curves were determined from a test of only 8 m. of yarn; submitting a greater yarn length to the test would very likely have led to a slightly smoother curve and possibly to a slight increase in the standard deviation measured.

Yarn Thickness Irregularity

Reference has been made to yarn thickness irregularity, based on weight or fiber number measurement; in order to avoid misunderstandings, the relation between standard deviation and test length shall be elucidated by a few additional remarks. For the sake of simplicity, irregularity tests based on weight measurement will be considered.

As an example, let us assume that the weight of many yarn pieces of 1 m. length has been determined; the mean value and the standard deviation of all the weights may be determined readily; the standard deviation is the square root of the sum of squares of the difference between the individual weights and the mean, divided by the number of weighed pieces minus one. The standard deviation thus defined considers the variation *between* the lengths of 1 m.

However, there is also a certain irregularity within the yarn lengths, which could be measured in the same way by cutting the lengths into very short pieces—*e.g.*, 1 cm.; the new standard deviation thus found considers the variation *within* lengths. The total irregularity of the yarn under control would obviously be characterized by a standard deviation obtained as the vectorial sum of the standard deviation between and within lengths.

For the discussion to follow, it is of importance how the two parts of the total standard deviation are influenced by the length of the individual pieces, which was arbitrarily chosen as 1 m. It is obvious that the standard deviation between the lengths will become smaller if the length is increased; on the other hand, the variation within the length increases with increased length. The total variation present in the yarn coincides with the variation between the lengths, if the length is reduced so much that the variation within becomes negligibly small. The total variation may also become coincident with the variation within the lengths, if the length is increased so much that the variation between the lengths becomes negligible [2].

These considerations also hold true if, instead of weight variation, reference is made to fiber numbers; indeed, fiber number and yarn weight are approximately or exactly proportional.

Instruments to Measure Yarn Evenness

Many instruments have been invented in recent years to measure yarn evenness; however, only a few of the particularly successful designs shall be discussed here.

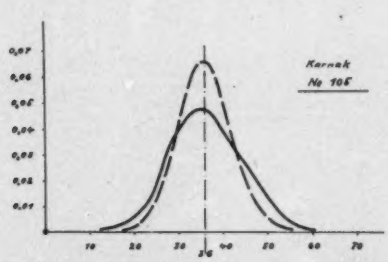

FIG. 2. *Cotton yarn Ne = 105. Fiber frequency distribution in yarn cross section.* Solid curve—*Experimental.* Dotted curve—*Theoretical, calculated according to Poisson law.*

TABLE I

Karnak cotton		Ne = 58	Ne = 105
Mean fiber number	n	67	36
Poisson standard deviation	s	8.2	6.0
Coefficient of variation (%)	$\frac{100}{\sqrt{n}}\%$	12.2	16.7
Coefficient of variation (%)	$\frac{106}{\sqrt{n}}\%$	13.0	17.7
Measured standard deviation	s^*	12.6	8.7
Corresponding coefficient of variation (%)	CV^*	18.8	23.6
Coefficient of variation read on integrator scale (%)	CV^*	17.9	22.4

* Measured values as opposed to calculated values.

The *Saco-Lowell sliver tester* was probably one of the first registering evenness testers to arouse interest and find wide application in industry. The very expedient and efficient design of this purely mechanical instrument is shown in Figure 3. The sliver or roving to be controlled is led through the calibrated groove of the driving wheel and compressed by a constant and known load; its cross section, measured under load, is registered greatly magnified on a chart and shown by a hand on a scale; the piece of sliver responsible for any individual reading is 2 to 3 mm. long. The instrument is designed for cotton slivers, and is suited for use in the laboratory as well as in the spinning mill.

Only very soft yarns, slivers, or rovings with no twist or very little twist can be tested with the Saco-Lowell tester, because only these adequately fill the rectangular section of the grooved wheel. With slivers and thick rovings, very reliable results are obtained. However, the instrument is not well suited for fine rovings and is not at all suited for finished yarns. In its original form, the apparatus evoked some prejudice because of the long and tiring work involved in the interpretation of the registered diagrams.

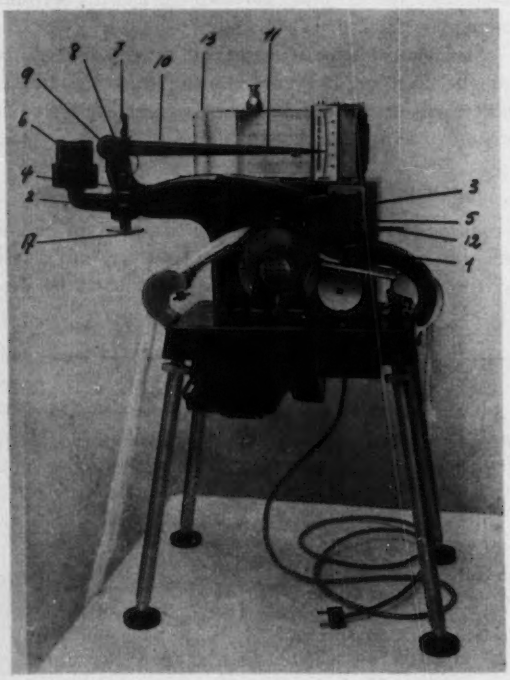

FIG. 3. *Saco-Lowell sliver tester.*

The *Uster evenness tester*, of more recent design, indirectly measures the cross section of the yarn: the yarn passes between the plates of a condenser and influences its electric capacity. The condenser is inserted into an electric oscillation circuit, and the changes in its capacity are measured by means of an amplifier; they can be read on a scale or registered on a chart. As shown in Figure 4, the yarn may be inserted in one of eight different slots, suitable for any thickness from a card sliver to the finest yarn. This tester may be used for vegetable, animal, and synthetic fibers. The capacity of the condenser is influenced by the volume of a piece of yarn approximately 8 mm. long; in almost all cases, the variation within this short length is negligible.

This instrument has wide application, because it admits various settings to serve different purposes. The speed of the yarn can be set at different values within wide limits; four different scales are available in the registering device, to suit the most divergent yarn irregularities; further, the registering device can be set for normal or slow reaction (inert setting), the first one responding to all, even the most abrupt, changes in yarn thickness, and the second one mainly to irregularities of long wave length.

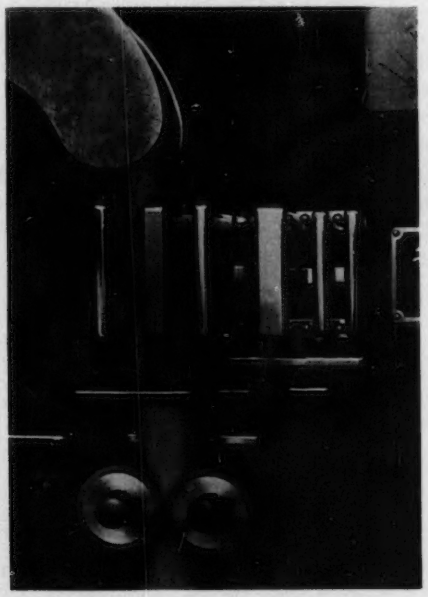

FIG. 4. *Measuring slots of the Uster evenness tester, showing a sliver being tested in the widest slot.*

While with normal setting the instrument registers the mean thickness of a yarn piece 8 mm. long, this length may be greatly increased by using the inert setting; the yarn length then considered by each individual reading depends upon the yarn speed, and may vary from 12 cm. for a yarn speed of 2 m./min. (40 times the chart speed) to 600 cm. for a yarn speed of 100 m./min. Used in this way, the instrument does not take into account the irregularity within 12 cm. and 600 cm. yarn lengths, respectively, and mainly registers the standard deviation between yarn pieces of these lengths. In accordance with the original weighing test, this length is designated the "equivalent cut length."

The efficiency of the instrument may be increased by using it in connection with an integrator, which automatically determines the mean of the linear or squared deviation for a given time interval corresponding to a certain yarn length. The apparatus determines the mean of the absolute value of the linear deviation; a similar instrument automatically determines the standard deviation. The standard deviation thus measured is that between lengths, neglecting the irregularity within the yarn lengths of 8 mm. The apparatus calculates the sum of the squares of the deviation from the mean for a determined time interval and shows the value on the scale; the yarn length, to which this value for the standard deviation refers, obviously depends upon the adopted running speed of the yarn. The standard deviation increases with the yarn length, to which it refers, and approaches a limit value asymptotically with increasing yarn length.

Strength tests of yarn may also be considered as indirect measures for evenness. Before modern testing instruments were available, the tensile test was used for this purpose, owing to its precision and reliability; even now its results may be of great help in forming a comprehensive judgment about yarn quality. It is, however, essential that a sufficient number of individual tensile tests be carried out and that the results be submitted to correct interpretation; a considerable number of tests is important in itself, as the control is extended to a greater yarn length.

The tensile test measures the force necessary to break the weakest part of the stressed yarn length; the stronger parts have no influence at all on the result. For research purposes it may be preferable to use short test pieces, perhaps only slightly exceeding the length of the longest fibers in the yarn, whereby the variation within the test pieces is very much reduced; accordingly, the variation between the tests is increased. Short test pieces are not often used, however, due to the fact that their elongation is not easy to measure. In many countries the standard length for single-strand tensile tests is 50 cm., or 20 in.

When the tensile test for yarn evenness control is used, a considerable number of individual tests are necessary. Automatic tensile testing machines prove particularly useful, because they automatically carry out any prescribed number of tests and prepare the interpretation of results up to a certain point. The *automatic tensile testing machine* unwinds the yarn from the bobbin, inserts it into the jaws, increases the load up to the breaking point, registers the breaking load and elongation, and also shows the sums of all loads and elongations; moreover, it automatically registers the load frequency curve. After completing the prescribed number of tests, the machine stops automatically. It also keeps a record of the sequence of load and elongation values (Figure 17).

The *continuous yarn tester* developed by Dr. Frenzel is particularly suited to finding the weak spots in a long length of yarn, a test to which great importance is attached in certain cases. Of late, the efficiency of the Frenzel machine has been decidedly increased by reducing the stressed yarn length and by introducing an electric stress control, free of inertia errors [10].

Some Examples of Test Results in Cotton Spinning

The practical importance of evenness tests will be shown by giving examples of tests carried out and the information derived from the results obtained. Cotton spinning was selected because of its great importance; however, similar examples from other branches of the textile industry could be mentioned as well; indeed, as far as evenness of yarn is concerned, the problems are very much the same, regardless of the quality and properties of the fibers.

Card Sliver of $1\frac{1}{16}$-in. Texas Cotton

The three diagrams in Figure 5 show the thickness variations of a card sliver Ne* = 0.2. In the three cases the scale is the same, from − 25% to + 25% deviation from the mean at the bottom and top limits of the chart.

* Ne = English cotton count.

In all charts the highest deviation registered is approximately − 10% and + 10%. Longitudinally, the three charts are completely different. In the top diagram the sliver speed was 8 times greater than the speed of the chart, and in the center one 32 times greater. The sliver length registered on the center diagram is therefore 4 times greater than on the top one, everything else being equal. The top diagram shows the slightest irregularity very clearly, while the center one gives a good general survey for a considerable length of sliver.

The bottom diagram is of entirely different character: the yarn speed in this case was 160 times greater than the speed of the chart and inert registering was applied in order to show only long-period irregularities; the numerous short-period variations observed in the first two diagrams are not registered here, but, on the other hand, thickness irregularities over great lengths are shown very clearly because the diagram refers to a sliver 5 times longer than the one above it. Whereas in the first two diagrams, made with normal setting, the equivalent cut length is 8 mm., in the bottom diagram, made with inert setting, it is approximately 120 mm.

Since the card sliver under consideration has approximately 15,000 fibers per cross section, its Poisson standard deviation is equal to 125 and the coefficient of variation, taking account also of the fiber thickness variability, is equal to 0.85%. The actual value of the standard deviation can be found by interpreting the curves registered on the diagrams, or directly when the test is carried out with the help of the integrator; the coefficient of variation thus found is equal to 3.55% with the normal test and 2.40% with the inert reaction test. These values, which obviously also take account of fiber variability, although they are considerably higher than the theoretical value, must be judged as favorable; it is remarkable that the ordinary standard deviation, taking account of all variations, is 1.5 times greater than the one on inert reaction, considering only long-period variations.

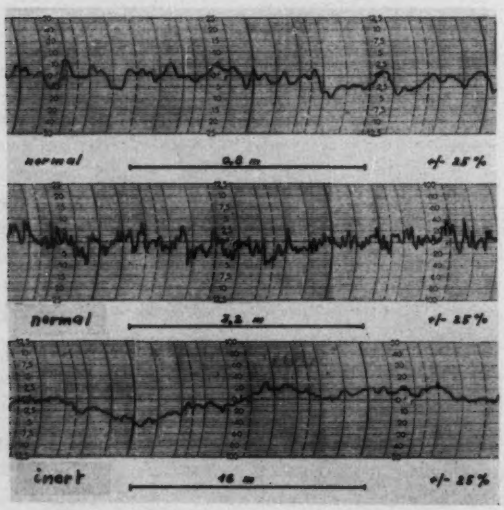

FIG. 5. *Card sliver Ne = 0.2 of 1 1/16-in. Texas cotton. Irregularity diagrams. (The length scale below the diagrams refers to the controlled yarn length.)*

Influence of Further Processes on Slivers

Slivers are processed on drawing frames in order to straighten and parallelize the fibers, and also to reduce sliver irregularities, particularly long-period variations which are likely to lead to count irregularities in the resulting yarn. The beneficial influence of doubling is particularly evident when processing combed slivers, as will be shown. Long and short fibers are not necessarily distributed evenly in the card sliver; consequently, combing will cause increased irregularity, as is strikingly revealed by the diagrams in Figure 6. The top two diagrams were registered normally, and the other two with inert reaction; in the top three diagrams the scale from − 50% to + 50% was used, and in the last one the scale from − 25% to + 25%. The yarn lengths are indicated on the diagrams. Data relating to these slivers are given in Table II.

As the top and the third diagrams show, the sliver is very irregular; its coefficient of variation was found to be approximately 13% by the normal test. The

TABLE II. SLIVERS OF 1 1/16-IN. TEXAS COTTON (COMBED YARN NE = 40)

Sliver	Tested	Speed ratio (chart: sliver)	Coeff. of variation (%)	Approximate equivalent cut length (cm.)
Combed sliver	normal	1:8	12.6	0.8
	inert	1:40	6.65	12
	normal	1:32	12.7	0.8
	inert	1:160	3.5	50
Sliver after two drawing frames	normal	1:8	6.35	0.8
	inert	1:40	4.15	12
	normal	1:32	6.25	0.8
	inert	1:160	2.9	50

inert tests were carried out using different sliver speeds, corresponding to equivalent cut lengths of 12 cm. (second diagram) and 50 cm. (fourth diagram); the coefficients of variation were found to be 6.65% and 3.5%, respectively. Compared to the card sliver, a general deterioration has taken place, being particularly pronounced in variations of short periodicity, as revealed by the top two diagrams.

After passing two drawing frames (draft and doubling both equal to 6), the evenness test of the sliver, carried out as before, leads to the four diagrams in Figure 7; in three of these diagrams the same scale, from +/− 12.5% variation, was applied, suiting the improved regularity. By the process, involving 36 doublings, the short-period irregularity was reduced to half and the long-period irregularity was reduced from one- to two-thirds of the previous values. The pronounced periodicity present in the bottom diagram has a mean wave length of 3.20 m.; this may have been caused by a wave of 9 cm. length in the combed sliver, generated either by the combing itself or as drafting wave by the subsequent drawing. Additional tests would be required to determine the origin of the irregularity with certainty.

As can be seen from Table II, the total irregularity was reduced to half by two drawing processes. Doubling d times theoretically reduces the standard deviation as follows [2, 8]:

$$s = \frac{s_o}{\sqrt{d}}.$$

Therefore, it ought to have been reduced to one-sixth by doubling 36 times in two drawing frame passages. The actual result does not compare well with the expected result, obviously because part of the improvement realized by doubling is cancelled by the imperfection of the drafting. The table and the diagrams show that long-period irregularity is also considerably reduced. The greatest improvement is in the evenness over great lengths, far exceeding the length of the drafting waves. Only evenness tester diagrams of corresponding length will show this kind

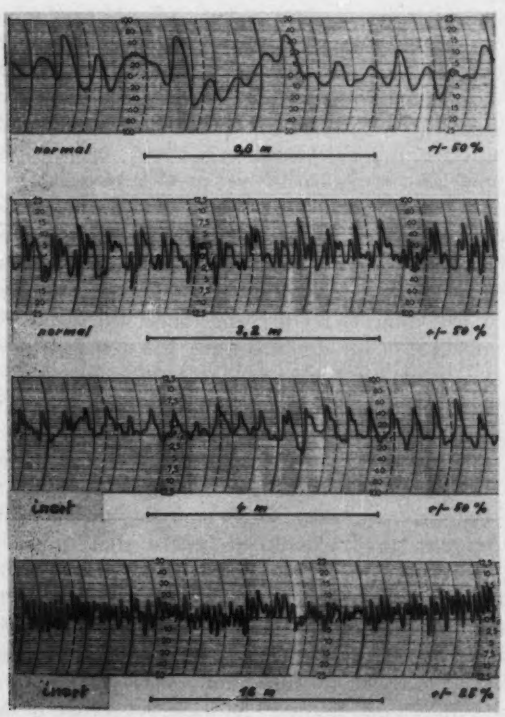

FIG. 6. *Combed sliver Ne = 0.18 of $1\frac{1}{16}$-in. Texas cotton. Irregularity diagrams.*

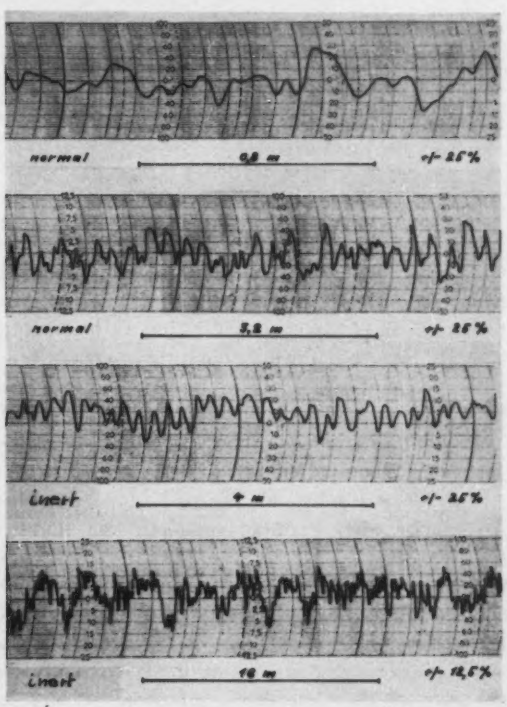

FIG. 7. *Sliver of Figure 6 after passing two drawing frames.*

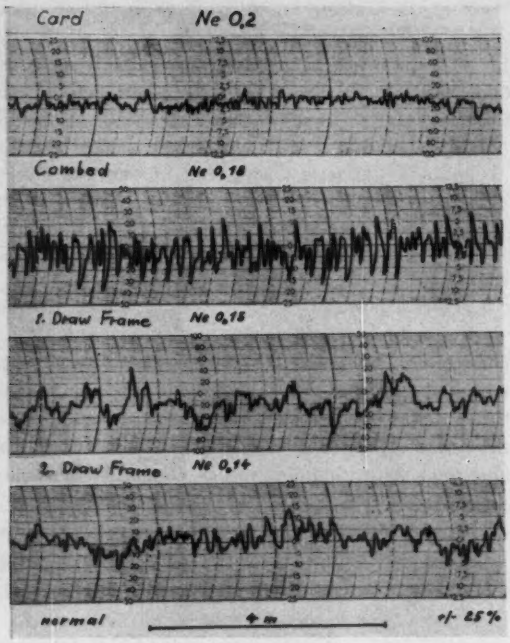

FIG. 8. *Spinning process, Karnak cotton yarn Ne = 76. Diagrams of card, combed, and drawing slivers.*

of improvement; the samples reproduced herein are too short for this purpose.

Ring Spun Yarn Ne = 76, Karnak Cotton

The evenness tester proves very useful in controlling yarn spinning through the various processes from the card to the ring spinning frame. By controlling finished yarns, which, owing to their greater strength, may be drawn through the evenness tester with increased speed, the equivalent cut length on inert reaction test can increase to 6 m.—*i.e.*, 20 ft. Tests of this kind will show only irregularities of particularly long periodicity.

The four diagrams in Figure 8 show the sliver irregularity after carding, combing, and the first and the second drawing frames. In all of these diagrams the yarn speed was 40 times the chart speed and the scale used was from -25% to $+25\%$. Further particulars, including the theoretical Poisson standard deviation, are given in Table III. The influence of the sliver-lap machine alone and the ribbon-lap machine alone, which are used after the card and before the comber, has not been tested.

TABLE III. SPINNING PROCESS (KARNAK COTTON YARN NE = 76)

	Ne	CV $(106/\sqrt{n})$ (%)	CV (measured) (%)
Card sliver	0.2	0.75	2.7
Combed sliver	0.185	0.70	5.4
First drawing frame sliver	0.135	0.60	4.5
Second drawing frame sliver	0.145	0.65	4.1
Slubber	0.65	1.30	6.5
Intermediate	1.45	1.90	6.2
Roving	4.70	3.50	7.1
Yarn	76	14.30	20.5

The regularity of the card sliver is superior to that of any following sliver. The sliver irregularity reaches its peak value after combing; as stated before, this must be attributed to uneven distribution of short fibers, to be eliminated as noils, and to the drafting wave of the drawing system at the end of the comber.

The first and second drawing frames reduce the sliver irregularity; however, as far as short-period variations are concerned, the degree of evenness of the original card sliver is not realized again. Two 8-fold doublings theoretically ought to reduce the deviation to a very small value, approximately identical with the Poisson limit. The actual improvement, as given in Table III, is very much smaller; this, however, is accompanied by a more important improvement of regularity over great lengths, which is not shown by the short diagrams. The divergence of experimental results from theoretical results may be attributed to imperfection of the drawing process, which is clearly revealed by the drafting wave. Accurate setting of the drawing frame will reduce the amplitude of the drafting wave, but it is impossible to eliminate it entirely. It is remarkable that calculus will tell us that a drafting wave may be generated in the drawing frame [4].

The influence of processing on the speed frames is illustrated by the three diagrams in Figure 9, which were registered like the preceding ones, but with the yarn speed reduced to half in order to make particulars more clearly visible. As shown in Table III, the coefficient of variation remains almost constant, in spite of the gradual reduction of cross section; this is to be attributed mainly to the roving twist and to the doubling taking place on the second and third speed frames. As the cross section of the roving becomes thinner, the length of the irregularity waves shows a tendency to become shorter.

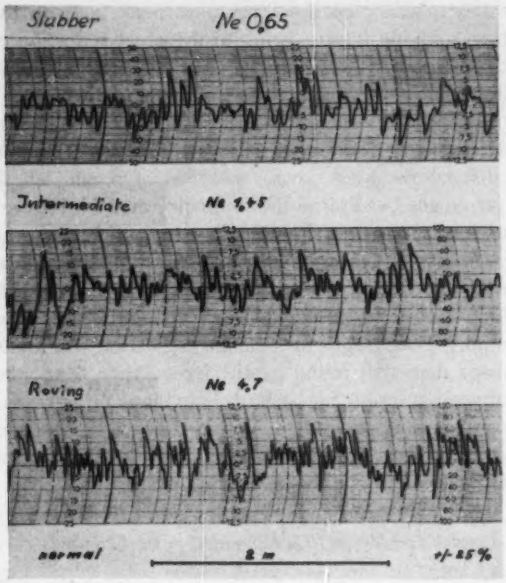

FIG. 9. *Spinning process, Karnak cotton combed yarn Ne = 76. Diagrams of rovings from slubber and intermediate and roving frames.*

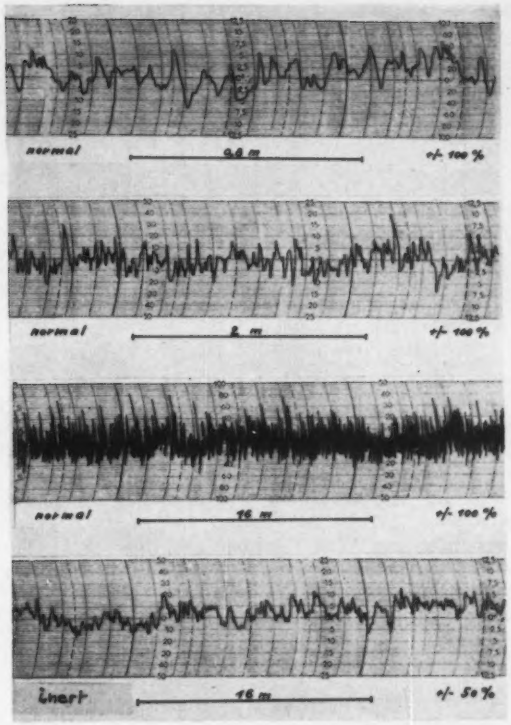

FIG. 10. *Spinning process, Karnak cotton combed yarn Ne = 76. Diagrams of the yarn itself.*

Particular attention was devoted to the test of the yarn itself. The top three diagrams in Figure 10 were made to the same scale, from − 100% to + 100%, but with different yarn speeds, increasing from 8 to 20 and to 160 times the speed of the chart. The third diagram is practically illegible; it is so compressed that its lines cover each other to a great extent. The bottom diagram was registered with the same fast yarn speed but with inert reaction, to take account only of long-period irregularities; in this case the coefficients of variation were found to be 7% with equivalent cut length of 12 cm., 5.6% with equivalent cut length of 150 cm., and 4% with equivalent cut length of 600 cm. (not shown in Figure 10). In the normal test (equivalent cut length of 0.8 cm.) the coefficient of variation was 20.5%. This value compares favorably with the theoretical value of 14.3%, which is for yarn with a mean fiber number per cross section of only 55. According to these figures, the quality of this yarn is superior to the average. The progressive development of the theoretical and effective standard deviation from the card sliver to the finished yarn is shown schematically in Figure 11. The actual standard deviation increases rapidly on the comber, on the

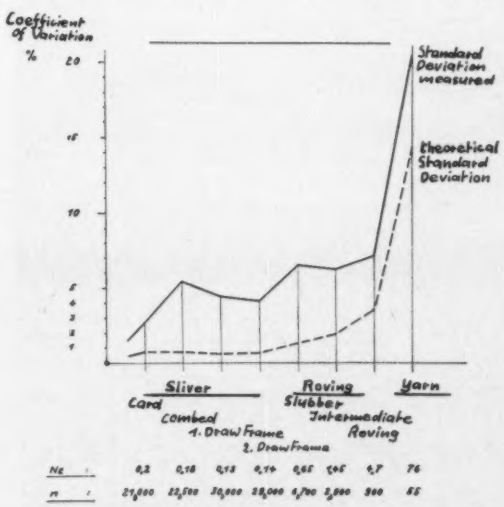

FIG. 11. *Schematic picture of the development of the theoretical and actual standard deviation from card sliver to finished yarn.*

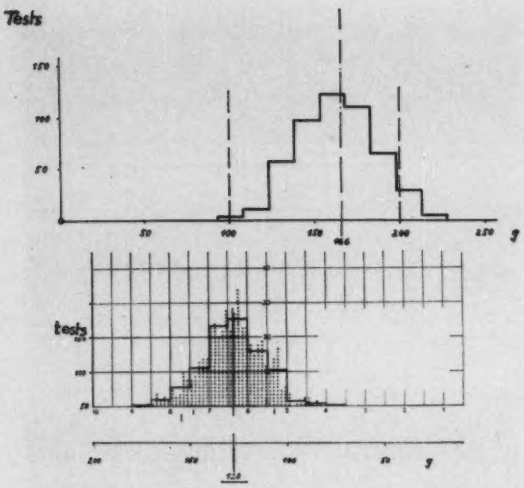

FIG. 12. *Karnak cotton yarn Ne = 76. Frequency distribution of tensile test results. Length of test pieces, 5 cm.* (top) *and 50 cm.* (bottom); *500 tests each. The dots on the bottom diagram were actually registered by the automatic tester.*

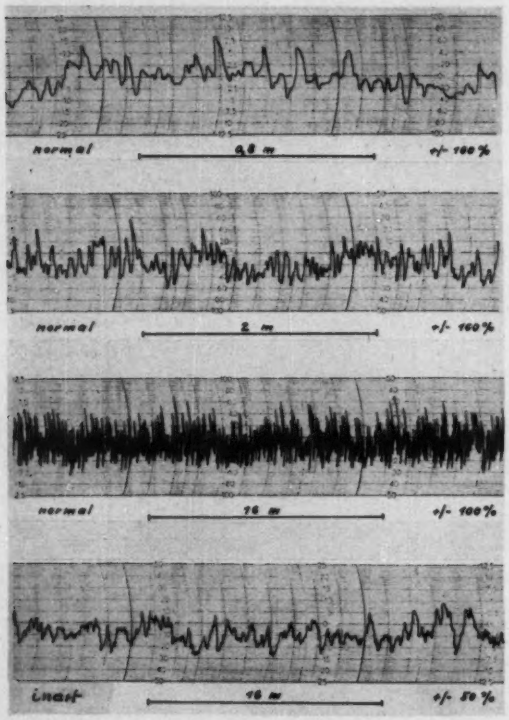

FIG. 13. *Evenness diagrams of Karnak cotton combed yarn Ne = 105.*

slubber, where no doubling takes place, and on the ring spinning frame, owing to the great reduction of cross section; accordingly, on the ring spinning frame the theoretical value also shows a steep rise.

The tensile strength of yarn from one bobbin was determined. Five-hundred tests of 50 cm. length (distance between jaws) and 500 of 5 cm. length were made, leading to the frequency distributions for strength shown in Figure 12. As anticipated, the tensile strength and its standard deviation determined on the short test pieces were higher than those on the long test pieces (Table IV). Even with test pieces of only 5 cm. length the standard deviation was much lower than that found by the irregularity tests; the same observation has been made in many other cases; it must be attributed to increased twist in the thin yarn sections and the accumulation of short fibers in thick parts.

Cotton Yarn Ne = 105, Karnak $1\frac{9}{16}$ in. Combed

This is an example of a fine yarn. The distribution curve for the fiber number per cross section in Figure 2 refers to this yarn. A yarn with a mean number of only 36 fibers per cross section is bound to have a considerable variation in thickness, since the Poisson standard deviation is as high as 17.7% of the mean; in reality, a considerably greater irregularity is to be expected, because spinning difficulties increase rapidly with yarn fineness. This is confirmed by Figure 2.

Accordingly, the scale -100% to $+100\%$ was used in registering the normal irregularity diagrams in Figure 13; in spite of that, the peak values are very far from the mean, in good agreement with the high value, 23.6%, of the coefficient of variation (Table V). The top three diagrams, registered with increasing speed, clearly show the character of the yarn irregularity in detail and also give a general picture for a considerably long length of yarn.

TABLE IV. TENSILE TEST RESULTS (500 TESTS) FOR KARNAK COTTON YARN NE = 76

Distance between jaws (cm.)	Mean breaking load (g.)	Breaking length* (km.)	Standard deviation (g.)	Coeff. of variation (%)
5	166	21.4	22.6	13.6
50	128	16.5	16.3	12.7

* Length of yarn, the weight of which is equal to the mean breaking load.

TABLE V. COTTON YARN NE = 105
(KARNAK $1\frac{9}{16}$ IN. COMBED)

Coefficient of variation $(106/\sqrt{n})$ (%)		*CV* total variation (measured) (%)		*CV* long periods only (measured) (%)
17.7		23.6		9.3
Tensile Tests (600 tests)				
Distance between jaws (cm.)	Bobbins	Mean breaking load (g.)	Standard deviation (measured) (g.)	*CV* (%)	Breaking length (km.)
5	1	92.8	17.5	18.8	16.5
5	10	95.8	22.9	23.9	17.1
50	1	87.0	14.2	16.3	15.5
50	10	76.6	17.3	22.6	13.7

The bottom diagram was registered with inert reaction and appropriate scale, leading to coefficients of variation for long-period irregularity of only 9.3% (equivalent cut length of 12 cm.) and 4.5% (equivalent cut length of 600 cm.) (not shown in Figure 13).

This yarn was also submitted to exhaustive tensile tests, the distance between the jaws having been set at 5 cm. and at 50 cm. for two different series of tests. The number of tests for one series was 600. The test pieces were taken alternatively from one and from ten bobbins (Table V and Figure 14). In agreement with theory and previous tests, the short test pieces led to higher values for the average tensile strength and its standard deviation. The difference between series of tests of yarn taken from one bobbin and yarn taken from ten bobbins was not conclusive as far as the mean is concerned; apparently, some random irregularity biased the results. However, the standard deviation showed a considerable increase when the test pieces were taken from ten bobbins; the fact that the coefficient of variation for tensile tests was as high as for the irregularity test is to be attributed, at least partly, to chance.

Cotton Yarn Ne = 58, Karnak $1\frac{9}{16}$ in. Combed

This last example was selected because of its remarkable degree of uniformity.

As in the previous cases, the normal evenness tests were carried out with three different yarn speeds, leading to the top three diagrams in Figure 15; a test with inert reaction was also made. The only difference between the top three diagrams is the length coordinate, varied as shown on the diagrams. The bottom diagram takes account mainly of long-

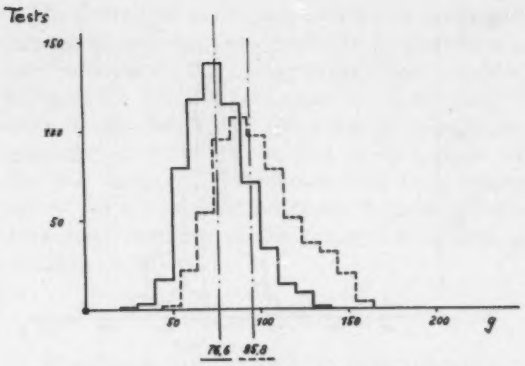

FIG. 14. *Karnak cotton combed yarn Ne = 105. Frequency distribution of tensile test results. Ten bobbins; 600 tests; length of test pieces, 5 cm.* (dotted line) *and 50 cm.* (solid line).

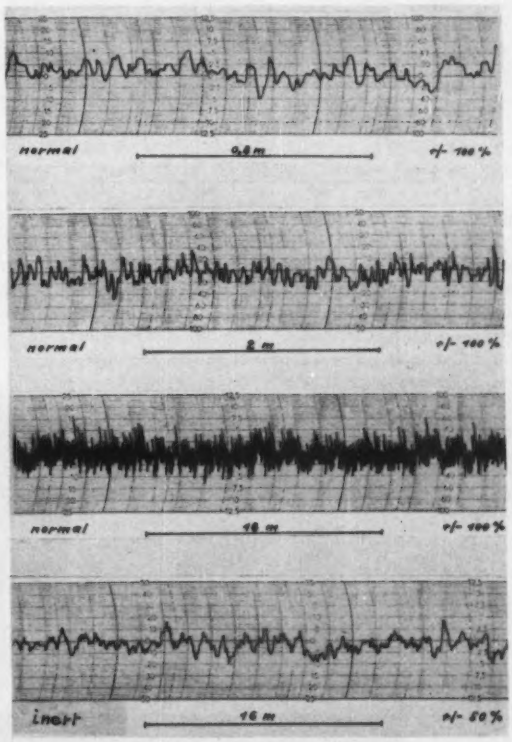

FIG. 15. *Evenness diagrams of Karnak cotton combed yarn Ne = 58.*

period variations, its equivalent cut length being 12 cm. If the yarn speed is increased to produce an equivalent cut length of 600 cm., the standard devia-

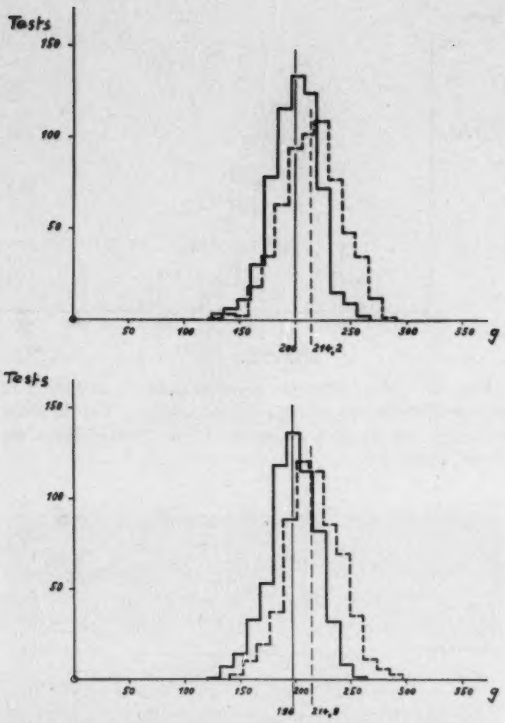

FIG. 16. *Karnak cotton combed yarn Ne = 58. Frequency distribution of tensile test results. Length of test pieces, 5 cm.* (dotted line) *and 50 cm.* (solid line). Top *—600 tests from one bobbin.* Bottom*—600 tests from 10 bobbins.*

tion measured on the evenness tester is as low as 3.3%.

The tensile tests were carried out on test piece lengths of 5 cm. and 50 cm., and the results are shown as frequency distribution curves for 600 individual tests in Figure 16. The top curves refer to test pieces from one bobbin, and the bottom ones to test pieces from ten different bobbins, 60 from each bobbin. Figure 17 shows the sequence of the in-

TABLE VI. COTTON YARN NE = 58
(KARNAK $1\frac{5}{16}$ IN. COMBED)

		Coefficient of variation ($106/\sqrt{n}$) (%)	Coefficient of variation (measured) (%)
Roving Ne = 5		3.8	6.3
Yarn		13	15.9
	Tensile Tests (600 tests)		

Distance between jaws (cm.)	Bobbins	Mean breaking load (g.)	Standard deviation (measured) (g.)	*CV* (%)	Breaking length (km.)
5	1	214.2	26.4	12.1	21.2*
5	10	214.9	24.3	11.3	21.2*
50	1	200.0	20.4	10.2	19.7
50	10	198.0	21.8	11.0	19.5

* In this case the standard deviation for tests from 10 bobbins was smaller than for tests from 1 bobbin. This was unexpected and is to be considered casual. It is important to note that the standard deviations for 1 and 10 bobbins are practically equal.

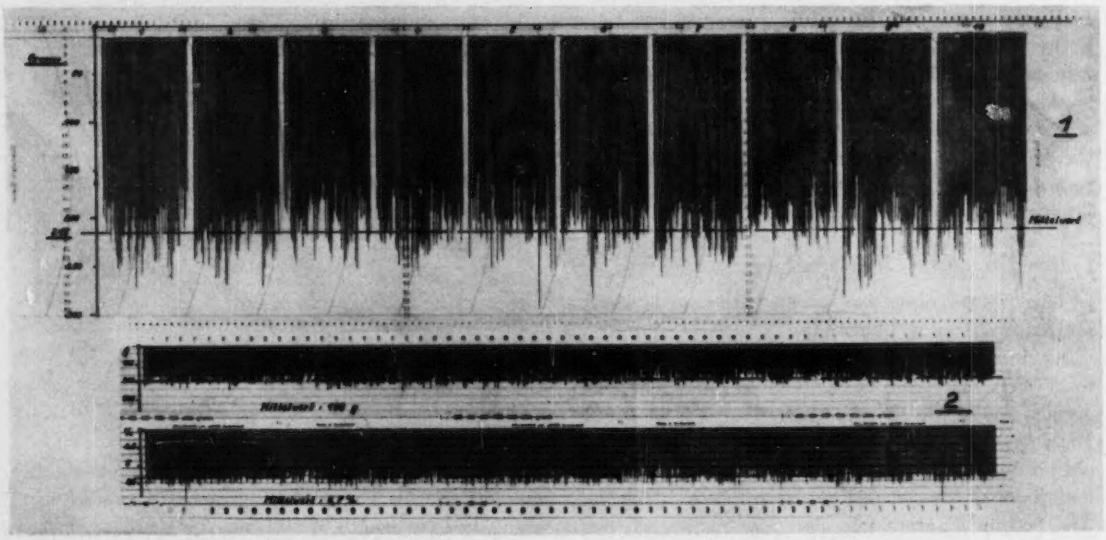

FIG. 17. *Sequence of test results.* 1*—Tensile strength; test piece length 5 cm.; 600 tests from 10 bobbins.* 2*—Tensile strength and elongation; test piece length 50 cm.; 600 tests from 10 bobbins.*

dividual tensile test results: *1* shows the breaking loads of the 5-cm. test pieces, each group of 60 tests referring to yarn from one bobbin; and *2*, not divided in groups and with different scale, gives the breaking loads and elongations of the 50-cm. test pieces, also derived from ten different bobbins. These diagrams, which were traced by the automatic testing machines, may in particular cases give valuable clues about the nature of irregularity.

These diagrams and the data given in Table VI indicate that the yarn tested shows a most remarkable degree of evenness; it is to be considered a splendid achievement of a spinning mill. The variations of the roving and of the yarn are very small, the average yarn tensile strength is remarkably high, and the standard deviation of the tensile strength is very low; moreover, the frequency distribution for the tensile strength of yarn from one bobbin and of yarn from ten bobbins is equal. In accordance with the extraordinary uniformity of the yarn, the influence of the distance between jaws on tensile test results is unusually slight [6].

Conclusion

The new automatic methods of testing yarn evenness and strength determine the fundamental properties of a yarn with a precision heretofore unknown and with a minimum of work. They make it possible to classify a yarn according to quality in a most reliable way. As shown by the various examples given, yarn evenness and tensile strength are closely related; as evenness improves, the tensile strength increases. The variation of yarn cross section obviously has an influence on the variation of tensile strength.

In many cases the irregularity diagram of an unsatisfactory yarn quality may give useful hints as to the probable sources of the deficiency.

The determination of evenness from the card sliver through all spinning processes to the finished yarn is most instructive. If the irregularity shows a sudden increase, for which there seems no justification, it must be concluded that either the process is not correctly set or the spinning plan is not suitable for the fiber quality. The evenness test is a reliable control for the spinning itself. No doubt it will also have great influence on the future development of spinning machinery.

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Irregularity in Textile Yarns and Strands

A Literature Survey

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AN INVESTIGATION into the measurement and causes of irregularities in textile strands formed the basis for this literature survey. Because emphasis was placed on the effect of textile processes on uniformity, the section covering the interpretation of uniformity measurements is quite detailed and the section describing measuring instruments is necessarily rather sketchy. For essentially the same reason, attention was given to the measurement of fiber parallelization, since this quantity has a very important effect on the uniformity of the strand after processing.

Measurement of Strand Uniformity

The problem of mathematically assessing the uniformity of textile strands involves the development of a means for measuring the linear variations of a strand property such as weight or diameter and also a method for interpreting the measurements once they have been made. This section is comprised of two parts, the first being a discussion of methods of measurement and the second being various workers' concepts on interpretation.

Measurement of Strand Properties

The archaic method of analyzing the uniformity of textile strands involves making a large number of measurements and tabulating data for further computation. One could measure a property such as diameter under the microscope, or, as Balls [6] did, use an air-flow instrument, in which a yarn partially obstructs an orifice through which air is flowing, the air pressure being considered an index of yarn area. Another, older method is to cut strands to the same length, and weigh the pieces. Martindale [48] described an aid for doing this which uses a yarn skein reel with a clamp on one arm so that a skein may be cut into several long and several short pieces.

Martindale [49] indicated one source of error connected with measurements made at definite intervals. If the strand has periodic variations and the period of measurement is related to the fundamental period of variation, then the measurements will be in error.

In order to speed up measurements, testers which produce a continuous record of a strand property were developed. The earliest type was the Saco-Lowell [82] tongue-and-groove compression tester. It has been used a great deal in its original form (see, *e.g.*, [43]) both in research work and in mills. It was modified by Rusca [65], who made the pen arm brush over electrical contacts, thus producing signals which actuate counters; and also by Germans [28] and Vose and Plummer [80], who constructed the integrators described below. Several British authors described similar instruments, including Thorndike [73] and Cavaney *et al.* [10]. The latter calibrated their instrument by cutting strands into $\frac{1}{8}$-in. lengths and weighing these. They stated that the effective length of contact is 0.1 in. on rovings, and somewhat more on slivers. Anderson *et al.* [2] described an instrument, probably the same as Cavaney's, in which the distribution of sliver thicknesses is found by having the pen arm move a water stream over a series of tubes so that the height in various tubes shows the distribution of strand thicknesses. The Pacific Tester [40] is a modification of the Saco-Lowell instrument, in which the top roll is connected to a strain gage, and the recording is electrical. Instruments of this type require the tongue-and-groove rollers to be machined to very close tolerances. For this reason, Steiger [70] built a simpler instrument, in which the sliver is pulled through a rectangular slot; a weighted top piece fits down into the slot, compresses the sliver, and is connected to a pen arm through a system of levers.

The photoelectric continuous evenness testers, of which two representative models are the S.E.R.C. instrument [69] and the Chamberlain Photometer [60], measure yarn silhouette. It has often been objected that the fuzz on a yarn causes errors in the measurement. The only workers who have

investigated the question seem to be Onions *et al.* [60], who reported that "gassing" the yarn (burning fuzz off) increases the measured variation slightly, but decreases the mean diameter much more than the measured weight loss would indicate.

In recent years the capacitance testers have come out. There are several of this type in common use, the I.T.T. Brush machine [4], the Uster tester [85], the Fielden bridge tester [83], and the Turl-Boyd tester [7]. These work on the principle of introducing a strand into a dielectric field. The change in capacity is (within limits) proportional to the amount of material. The theory is expanded well by Boyd [7]. While these instruments are quite sensitive to the amount of moisture in material, Walker [83] studied the effect of moisture and showed how to minimize the resultant error.

There are two miscellaneous types of testers which seem to be important. One is the W.I.R.A. (British) shoe tester [2, 10], in which yarn is pulled under a slightly weighted foot, the movement of which is transmitted to a photographic recorder. Cavaney *et al.* [10] found a correlation coefficient of 0.95 between these measurements and weights of short lengths. Using a much different type of test, Stout [71] examined the uniformity of yarns by winding at constant tension and counting the number of breaks. He covered both the theoretical and the experimental approaches.

To speed up the statistical computations, integrators have been devised for these continuous testers. Vose and Plummer [80] connected a Saco-Lowell tester to a variable transformer. The electrical signal is amplified and measured by watt meters which give the sum and sum of squares of observations, thus speeding up computation of the standard deviations. Germans [28] described an instrument called the Vitno-Integrator. The measuring device actuates a mirror galvanometer. A light beam, deflected by the mirror, passes through a mask which determines the action on the observation (summing, squaring, etc.) and onto a photocell. A condenser is initially charged, and is discharged by the current through the cell. The change in voltage, when read by an electrostatic voltmeter, becomes a measure of the integral. This instrument was also used with a Saco-Lowell tester, in which the pen arm is attached to the mask. Matthew *et al.* [52] described an electrical integrator which gives mean deviation from the mean. An article by Mullen [58] discusses the integrators which are available for commercial instruments in this country, and describes the action of each upon the measured quantities.

There is a very limited amount of literature available on the comparison of various types of instruments. However, Matthew *et al.* [52] gave an extensive review of the methods of measuring the irregularity of rovings and yarns, with a bibliography of 55 references. Also, the W.I.R.A. [84] and Townsend [76] reported on two series of tests made in 1947 and 1948 on worsted yarns. Considerable data were given, and an investigation of what property the various instruments measure was made. An article by Hasler and Honegger [36] gives some details of the Saco-Lowell and Uster testers and their use in tracing machinery defects. Also, an article by Mullen [58] gives a very comprehensive survey of the testers currently on the market in the United States, with the common accessories which may be obtained with them. The American Society for Testing Materials Committee D-13, under Subcommittee B-1, organized a Task Group for the Evaluation of Evenness Testers. This group, in its first report [4a], discusses an interlaboratory test in which worsted yarns were run on several instruments. The evenness charts were then evaluated by the group. They concluded that all of the testers studied measured a yarn property which could be correlated with weight per unit length. Included in the report is an extensive literature survey on instruments for measuring yarn evenness which has 55 references not mentioned in this paper.

Interpretation of Uniformity Records

The old, established method of evaluating the uniformity of a yarn is the visual examination of it as wound on black boards. Comparison with recognized standards or within-series comparisons are relied on for ranking, and in a well-organized system can give quantitative data of perhaps surprising accuracy and reproducibility. The scoring is subjective, however, and basic mass unevenness tends to be obscured by other factors, such as neps, fuzziness, twist, and trash, and their pattern of distribution. With this visual examination, these other factors are frequently made part of the rating as an important part of the over-all evaluation of the yarn.

Evaluation by sensing and recording instruments is usually done to measure mass unevenness alone, and efforts are made to minimize the effects of the other factors. In fact, it is a common belief that the most desirable instrument would be that which

records only the mass distribution. Thus, instruments do not tell the whole story about a yarn, and they are different in the proportions by which various factors influence their sensing elements. In some cases, discreet irregularities such as neps can be detected in the charts by characteristic pips. Instrument evaluations, with their permanent charts, do have distinct advantages in allowing mathematical interpretations, and many systems are adaptable for coarser strands than yarn.

The most important point in deciding which of the many approaches to employ in evaluating the charts is consideration of the use to which the measurements will be put. Two possible applications are the interpretation of unevenness in terms of end-use of the yarn and in terms of the processing history of the strand. In the first case, one would be interested in factors which control processability into the end-item, and those which govern fabric appearance, such as neps, slubs, and irregularities which would give rise to filling bars and other patterns in the cloth. In the latter case, one is interested in evaluating the performance of the various machines; this application is described fully in a later section. The methods described below are useful in either type of analysis.

Because of the various means by which unevenness may be measured, and various objectives or points of view as to the basis for evaluation, there is really no fixed and universally applicable measure of "unevenness." Use of this term, and any mathematical expressions for it, should always be given with clear reference to the methods employed.

This section discusses the various approaches which have been proposed, in the historical order of development. First, workers attempted to measure the period of the wavelike variations which occur along the strand. Then followed the measurement of amplitude variation and the relation between variation and the length within which it was measured. The most recent development has taken into account the theoretical random variation, which increases as the strand becomes finer.

To the British, it has seemed obvious to attempt to measure the period of these waves ever since Balls [5] coined the term "drafting wave." There have been two methods of accomplishing this. The first was developed by Foster [21] and Martindale [46], who described the "Grating Periodograph" and "Correlation Periodograph," respectively. These appear to be the same type of instrument. In each, a continuous record of a property (*e.g.*, weight) of a strand is prepared, and the instrument analyzes the record for the fundamental period and harmonics. Cox and Townsend [16] described the Correlogram method, in which the correlation coefficient between points a period L apart on a continuous record is measured. The correlation coefficient is then plotted against L. A pure periodic curve would be as shown in Figure 1a. A damped wave, as shown in Figure 1b, is usually found for textile strands. They believe the Correlogram to be the best method of detecting quasi-periods in strands. Methods of obtaining Correlograms include the optical correlographs (probably the instruments described by Foster and Martindale), computation by punched cards, and other tedious techniques.

Results of measurements show that the average fundamental drafting wave length in cotton yarn is about 2.5 in. [23]. For one particular yarn, Foster [22] expressed the measured wave lengths as harmonics of 84.9 cm. There seem to be about 20 separate waves, from the 4th to the 31st harmonics, making up this composite wave. In the further interpretation of the meaning of these wave lengths, Foster and Martindale [27] measured the fundamental wave length of the spinning drafting wave by the Correlogram method as a function of draft and roller setting, and found

$$\lambda = aD(h - b) + ch + d,$$

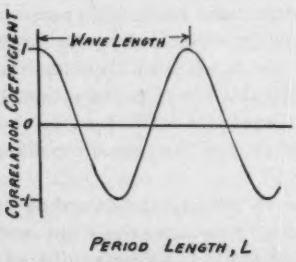

FIG. 1a. *Correlogram for a periodic curve (from Cox and Townsend [16]).*

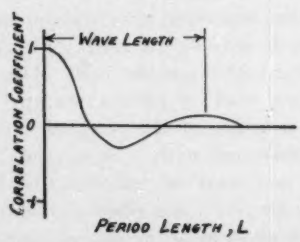

FIG. 1b. *Correlogram for a typical textile strand (from Cox and Townsend [16]).*

where a, b, c, and d are constants, λ = wave length, D = draft, and h = roller setting. The coefficient b is very close to the average staple length for normally twisted rovings.

The coefficient of variation (CV) has been found to be a very useful measurement in characterizing a strand. If the strand is considered to be composed of short elements placed end to end, the CV of the weight (or other property) of these elements is a measure of variability. For practical purposes, a quicker way of finding the CV than reading values off the charts must be found. The best way is to use integrators and other instruments which give processed data (means, sums of squares, etc.), which were described earlier.

A method for approximating the standard deviation was described by Temmerman and Hermanne [72] and Quintelier and Hermanne [64], who reported that they got the method from van den Abeele. On a Uster chart, lines are drawn parallel to the length axis at various ordinates. For each line on the higher than average side, the length of the strand which has a weight higher than the line is summed. Similarly, on the low side the total length having weight less than the line is found for each line. Then, using the property of the normal distribution which states that 34% of the area under the curve lies between the mean and the mean plus one standard deviation, the lines are found which have 16% of the length greater and less than each line, on the high and low sides, respectively. Then, the distance between the lines is two standard deviations.

A similar method was worked out in the Bates Laboratory by Perry [61], who assumed that each "pip" or deviation above (or below) the lines mentioned above occupies the same length. He prepared a series of charts relating the distance between lines and the number of pips to the standard deviation. Test performed by an outside laboratory, in which a thousand points were tabulated from a chart, corroborated this method of test.

A good discussion of the theory of the various methods of reading uniformity charts was given by Chakrabarti [11]. He calculated the CV by tabulating points, the mean deivation method, the mean range method, and the probit method (similar to those above). The mathematical derivation of the various methods was given. Mullen [58] described the methods of interpreting charts which are in common use in this country. He also discussed the types of measurements which the various commercial integrators give.

The relation between CV and the length of the period measured has been found to be very useful. Thus: if $V(T)$ is the CV of all the elements in the total length of a strand, if $V(L)$ is the CV of the elements in length L, and if $B(L)$ is the CV between means of strands of length L, then

$$V(L)^2 + B(L)^2 = V(T)^2.$$

Therefore, plots of V^2 and B^2 against L for a given yarn complement each other. This type of curve may be used to determine the optimum length of strand to test so that a good estimate of total variation may be made, as Elting and Barnes [18] did. On the other hand, Simpson *et al.* [67] wanted to test a short length, not affected by long-term variation. By using these curves, they selected for test lengths 1 yd. of sliver and 5 yds. of roving, and quoted Townend and Chow [74] and Cox and Ingham [15] in support of their choice.

The determination of the $V(L)$ and $B(L)$ curves can be quite tedious. The obvious method of finding the $B(L)$ curve is to cut various lengths and weigh them and calculate the CV. Lund [45] did this, weighing lengths from 1 in. to 120 yds., and cited the need for an automatic integrator for an uniformity instrument which would speed up the work. Townsend [77] reported that once one has prepared the Correlogram, the $V(L)$ curve can be found by simple integration. The best method seems to be a combination of reading continuous records and weighing lengths of strands, according to Townsend [75]. He used a continuous tester to obtain $V(L)$ for lengths up to 5 yds. Lengths of 5 yds. and longer are weighed, and $B(L)$ for these is calculated. From the equation

$$V(L)^2 + B(L)^2 = V(T)^2,$$

$V(T)$ can be found if $B(L)$ and $V(L)$ are in the same units. Townsend and Cox [77] analyzed several yarns and reported both curves and data.

The ultimate in uniformity is not a perfectly uniform strand, as one would at first think. Martindale [51] stated that since the fibers come off the card in a random order and since none of the succeeding processes do anything to even out irregularities, the ultimate in uniformity under the present system is a random sequence of thick and thin places. In order to compare actual variations with this "ideal" strand, workers have been constructing "ideal" mathematical models of textile strands.

Since the actual variation is always greater than the "ideal," no one has yet proved the mathematicians wrong. These workers, Spencer-Smith and Todd [68], Martindale [48], Huberty [39], Temmermane and Hermanne [72], van den Abeele [78], Olerup [59], Picard [62], and Breny [8], and probably others, generally worked from the fiber length distribution and, assuming random placement of fibers along the strand, worked through the Poisson distribution, and came up with an expression which states in a complicated way that the expected CV of strand weight is inversely proportional to the average number of fibers in the strand. Martindale [51] stated it rather simply in

$$V^2 = \frac{(100)^2}{N} + \frac{V_m^2}{N},$$

where $V_m = CV$ of fiber weight/length, $V = CV$ of yarn weight/length, and N = average number of fibers in a strand. For most cottons,

$$V^2 = \frac{(106)^2}{N},$$

$$N = \frac{15{,}000}{CF},$$

where F = fiber fineness (μg./in.), and C = cotton count.

The comparison of the actual variation to this ideal is the next logical step. Huberty [39], as reported by Temmerman and Hermanne [72], proposed a parameter

$$K = \frac{V\sqrt{N}}{100} = \frac{V}{100}\sqrt{\frac{\text{yarn denier}}{\text{fiber denier}}}.$$

Monfort [54] proposed a parameter I_r, which is similar to Huberty's K.

Workers in this country have developed similar ideas with range. Enrick [19] calculated the theoretical range:

$$R = 0.816\sqrt{\frac{\text{yarn number}}{\text{fiber fineness } (\mu\text{g./in.})}},$$

where R is the irregularity as measured on a linear tester. Casby [9] and Chandler [12] made similar analyses, and found parameters

$$B = U\sqrt{W},$$

$$C = \frac{U}{\sqrt{Y}},$$

where W = weight (grains/yd.), Y = yarn number, and U = evenness (%).

To speed up the reading of charts, some use the range method (reading spread between maximum and minimum in a given period), which is not as accurate as the CV can be. Rusca [66] compared range values with planimeter measurements, and found that the two methods usually class a series of yarns in the same order. He used range measurements for routine use because of their speed.

Many of the calculations, methods of finding CV, etc., assume that the distribution of weights of elements is normal. Apparently, the only one to investigate the assumption was Löfgren [43], who read about 1,500 points from uniformity charts for each of several types of rayon slivers. The card sliver he measured was skewed positively, but after drawing and doubling the frequency distribution was very close to normal.

A good summary of the work on this subject in the year 1950 was given by Anderson [1].

This section has covered, in a more or less historical order, the development of methods of interpreting uniformity charts, from attempts to measure wave length, through the coefficient of variation, and finally the comparison of measured with random variability. The concepts which seem to have the greatest value are the relationship between variation and length, and the comparison of actual with random variability. In consideration of the time involved in calculating CV's, it would seem that more use could be made of the range in variation-length relationships than has been to date.

Measurement of Fiber Parallelization

This section has been included in the survey because Löfgren [43] showed that the degree of parallelization of the fibers in a strand governs the way the strand responds to drafting. A highly parallelized strand drafts well; a poorly parallelized one yields a very irregular product on drafting. The methods of measuring this parameter fall into three classes: (*1*) those which measure fiber length; (*2*) those which employ friction or strength tests; (*3*) those which use tracer fibers in the strand.

The methods which measure fiber length in one manner or another seem to be accurate when the fibers are not very parallel but fall short in the later stages of parallelization. Hayman [37] in the Bates Laboratory did some work on a technique in which a sliver is cut to a length of 1 in. or $\frac{3}{4}$ in. The cut section is sorted on the Suter-Webb, and the weight of fibers over the cut length is expressed as a fraction

of the total weight. Since the Suter-Webb measures lengths in $\frac{1}{8}$-in. intervals, it is probably too crude for a finer work. Lindsley [42] tried the above test, but rejected it because of the time involved in sorting the fibers.

Lindsley then developed a method in which he clamped a sliver, cut it off $\frac{1}{2}$ in. from the clamp, combed the fibers out straight, cut off the extended tail at the $\frac{1}{2}$-in. point, and then weighed: portion removed by combing, C; extended tails cut off, E; portion remaining between clamp and $\frac{1}{2}$-in. line, N. Then, the combing ratio, $C/(E + N)$, depends upon fiber length, and the orientation index, $100(1 - E/N)$, varies from 87 for card sliver to 96 for first draw and 98 for roving.

Like Hayman's method, it seems that this test would be insensitive in the later stages of processing. Foster [26] described two methods, which probably also have this shortcoming. In the first, a bar is placed on a sliver, and the tufts are combed out on either side. The total weight of the tufts is M_1. The weight of sliver under the bar is M. Theoretically,

$$\frac{M_1}{M} = L + \frac{\sigma^2}{L},$$

where L is the average fiber length, σ is the standard deviation of fiber length. The experimental ratio of M_1/M divided by the theoretical gives the degree of parallelism, P_1. This value varies from 0.69 for card sliver to 0.84 for first drawing, and 0.86 for second and third drawing. His second method utilizes a bar, as above; he combs out the tuft on one side, cuts a strip of length b next to the bar, and finds its weight M_2. Then,

$$P_2 = \frac{M_2}{bM}\left(1 + \frac{b}{2L}\right).$$

No data were given for this method. Vogler [79] used a test similar to Foster's first method. He called it the Litty "Parallex" tester. The tufts and the portion under the bar are weighed on a special balance, which indicates the degree of parallelism.

It seems logical that a measurement of "drafting force," or the force necessary to part an untwisted sliver, would be a measure of the effective degree of parallelism. Thus, one could use an instrument such as Martindale [50] used for the measurement of drafting force while drafting is going on. This is described later. Löfgren [43] made a literature search on the various workers who studied interfiber friction. He developed a test in which an untwisted sliver is "broken" and the clinging force per fiber in the strand is calculated. If C_o = clinging power in card sliver, C_1 = clinging power in strand of highly parallelized fibers, and C = clinging power in strand under test, then the degree of parallelism may be expressed as

$$P = \frac{C_o - C}{C_o - C_1}.$$

Other workers who have measured interfiber friction (besides those mentioned in Löfgren's review) are Waggett [81], who studied the effect of finishing oils on the processability of rayon, Anderson *et al.* [3], and Cox and Ingham [15], who pulled single fibers from slivers.

The third method of finding the degree of parallelization of fibers in strands is to introduce tracer fibers which can be followed in some way. Morton [55, 56, 57] immersed the strand of cotton or rayon in a liquid of the same refractive index (tricresylphosphate) as the fibers, and then observed the position of dyed tracer fibers. He classed the fibers according to the type of hooks, and the fiber extent in the strand direction. Drafting increases the fiber extent and removes leading hooks more easily than trailing hooks. Also, hooks are formed when the strand rubs against stationary parts of the machine.

Effect of Textile Processes on Uniformity

The studies on the effect of textile processes on textile strands fall into two classes. The first is on the effect of normal processes (drafting and doubling) on strand properties, and the second covers the effect of defective machinery on the strands. The first class starts with rather superficial works which compare various types of processes.

In the field of cotton processing, Lee [41] was one of the early workers. He weighed 6-in. lengths of sliver, and found that drawing improves uniformity. Loveless [44] also studied drawing by putting sliver through drawing 33 times. The fibers became very parallel, and, in comparing yarn having 33 drawings with that having 3 drawings, it was found that the Uster evenness was reduced from 98% to 70% (discounting neps in yarn) and the yarn strength increased about 20%.

In studying the high-draft "short" processing of cotton, Quintelier [63] compared Casablancas roving with the old two process, and found improvement in roving, but this gain was lost in spinning. Mody [53] gave considerable discussion to Casa-

blancas spinning, but gave no comparative data on uniformity. Also on high-draft roving, Corley and Simpson [13] found the best ratios of back draft to total draft for various weights of sliver to produce roving of optimum evenness.

While similar studies on worsted processing do not correspond in detail to cotton, the general conclusion that high draft is injurious and that doublings tend to smooth out irregularities does apply, in substance, to cotton. Workers in this field are Martindale [47], Townend and Chow [74], and Cox and Ingham [15].

Martindale [50] made a detailed investigation of the force required to draft a sliver. His paper describes the instrument in detail and gives data. He reported that the variations in force are wave-like, very much like the variations in thickness of the drafted strand. The force varies directly with the weight of the element entering the draft zone and inversely with draft and roller setting, and is independent of the speed of drafting. The force is about four times as great for a card sliver as for a third-head drawing sliver.

The effect of draft and doublings upon yarn skein strength (which is more or less proportional to uniformity) was studied by Fiori [20]. He found that high spinning drafts decrease yarn strength and that the amount of doublings and draft in drawing, roving draft, and number of drawing processes have no effect on yarn strength.

An exhaustive study of the effect of various systems on uniformity was made by Lund [45], who spun rayon staple of various lengths and deniers on eight different systems (cotton ring, worsted cap, woolen condenser, etc.). The irregularity curves, $B(L)$ *vs.* lengths, were found by weighing lengths of yarn from 1 in. to 120 yds. Transmission prints of fabrics were presented. He discussed the data, and concluded that a single short-term uniformity test at a certain period and a single long-term test at another period are not adequate to assess the uniformity of a yarn; measurements at intermediate periods are needed.

The $B(L)$ and $V(L)$ curves were also used by Townsend [75], who used the worsted system to study the effect of draft and doublings on uniformity. The curves for three particular yarns are very interesting (Figure 2). *A* was produced with normal doublings and spinning draft, *B* had normal doublings but a very high spinning draft, and *C* had no doublings and a normal spinning draft. When woven into fabrics, *A* was satisfactory, *B* had streaks about 5 cm. long, and *C* had filling bars. He concluded that measurement of uniformity at only one period is unsatisfactory.

The measurement of uniformity was extended to use Huberty's comparison with random arrangement (K) by Temmerman and Hermanne [72]. They found the $V(L)$ and $B(L)$, and from them calculated K (internal) and K (external), respectively. They found that the draw frame increases K (int.) and decreases K (ext.) so that K (total) is about the same. They called the draw frame a parallelizer and changer of wave length. The roving frame keeps K almost constant, and spinning decreases K a little.

These authors considered that fibers move in groups rather than individually. For each stage of processing they calculated the number of fibers in a group on the condition that K be equal to unity. This number reduces from sliver through yarn.

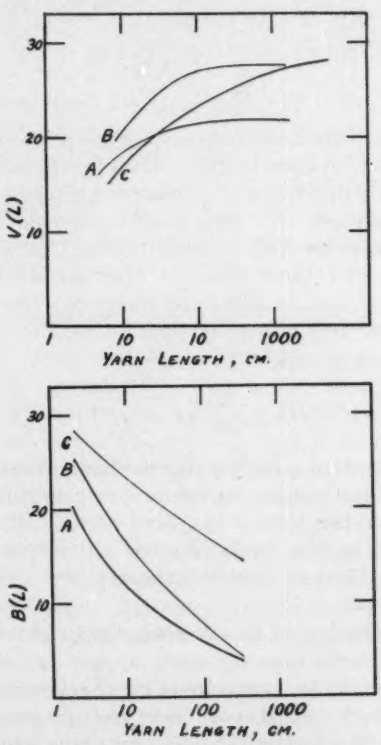

FIG. 2. *Worsted yarns.* A—*Normal drafts and doublings.* B—*High spinning draft, normal doublings.* C—*Normal draft, no doublings.* (*From Townsend* [75].)

After investigation of the effect of processes on uniformity, the next logical step is to attempt to formulate laws which govern the operations in normal textile processes—*i.e.*, doubling and drafting. The law of doubling is the more straightforward of the two. Theoretically, if n strands each of coefficient of variation V are doubled, the resultant coefficient of variation is

$$V_d = \frac{V}{\sqrt{n}}.$$

This relation has been found to hold true in practice.

The laws of drafting have been more difficult to formulate. If drafting is considered perfect, the resultant strand is geometrically similar to the ingoing strand. Elting and Barnes [18] proposed a performance ratio based on this conception. With perfect drafting, the resultant coefficient of variation can be found from the law of doubling. The ratio of this CV to the observed CV is termed the "performance ratio."

Simpson *et al.* [67] went a little further into the subject. In addition to proving the law of doubling, they studied the drafting of slivers into rovings, and found that a plot of roving CV against draft is parabolic. Also, for slivers of different weight, the slope of a semilog plot is inversely proportional to sliver weight. When strands of different weight are produced, the CV's must be compared on the basis of equivalent weights. The CV's are proportioned inversely with the square roots of weights. The authors found these techniques very useful for the prediction of product variability, so that drafts can be arranged to give a product of optimum evenness.

Löfgren [43] made a very comprehensive study on the processing of rayon staple. From a theoretical standpoint, he found that the irregularity ratio, E (ratio of resultant relative range or CV to that of the ingoing), is equal to

$$E = \frac{V_2}{V} = \sqrt{D(1 - B)},$$

where D is the draft, and B depends upon the fiber movement, structure of the strand, etc.

His greatest contribution was in showing the importance of having fibers parallel. The test for parallelism is outlined in an earlier section. In studying the influence of the roller setting distance, he found that when fibers are not parallel, great care must be used in obtaining the correct setting distance. Likewise, in studies of draft, when the fibers are not parallel, high draft is very injurious. He showed curves of irregularity plotted against draft, both theoretical and experimental curves, as shown in Figure 3. It is seen that for a low degree of parallelism, the experimental curve rises much steeper than that for the high degree of parallelism and the theoretical curves.

He also studied the effect of fiber length distribution, and made fiber mixtures having nine different fibrograms. He concluded that the fibrogram shape is very important when parallelism is low, and that the spinning ability of short fibers cannot be improved by adding a few long fibers.

The most important investigation into the drafting of cotton was made by Foster [23]. To aid in his investigation, he introduced the concept of added variance. If V is the coefficient of variation of the elements making up a strand, then, by the analysis of variance, the relative variance $(V_y)^2$ of a yarn is the sum of the variations introduced in spinning, roving, and other processes. Mathematically,

$$V_y^2 = V_s^2 + V_r^2 + V_d^2 + V_e^2,$$

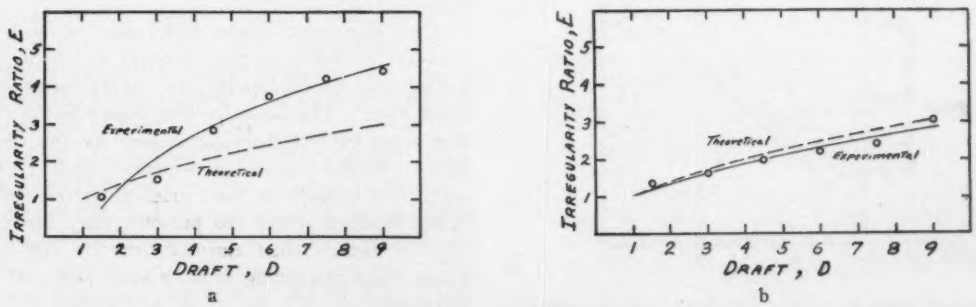

FIG. 3. *Irregularity ratio* vs. *draft.* a—*Low degree of parallelism.* b—*High degree of parallelism.* (*From Löfgren [43].*)

where the subscripts refer to spinning, roving, drawing, and card sliver, respectively. The sum of the last three terms is the relative variance of roving, so that if the total measured relative variance of roving is subtracted from the total relative variance of yarn, the result is the variance added in spinning. The law of doubling in this notation is simply: The relative variance of n strands doubled together is the relative variance of the singles divided by n.

He investigated the spinning of yarns from various weights of roving, and plotted added variance against draft, obtaining straight lines which converge to draft of unity at $V^2 = 0$. With finer rovings the slopes are steeper. Also, the added variance for a given draft is approximately proportional to the roving hank number. The graph is shown in Figure 4.

In addition to the variations caused by random placement of fibers and the drafting waves, there are often machinery defects which cause a great deal of trouble. Thus, Hannah [34], in working with the Ambler Super-Draft system, found that the design of rollers is much more critical at high drafts than at low drafts. Another source of trouble in the cotton draw frame was found by Dakin *et al.* [17], who found that improper weightings on rollers cause slippage and a very high amplitude wave in the product.

A more involved type of trouble was found by Gregory and Tyson [32], who studied the movement of the front-roll nip caused by worn bearings, eccentric rolls, etc., and also torsional vibrations in long rolls driven at the end of the roving frame, which cause speed variations. This work was also described by Foster [25], who stated that because mechanical imperfections such as these produce a variation in the number of leading fiber ends, if the strand is reversed before the next operation so that trailing ends are presented to the nip in the next drafting process, the defect is minimized.

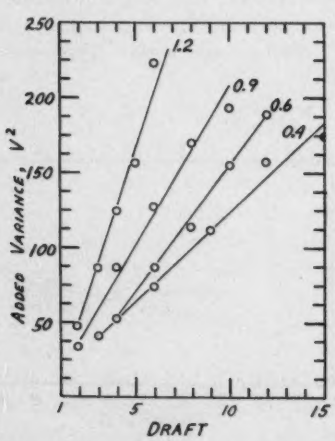

FIG. 4. *Added variance* vs. *draft on the intermediate frame. Numbers at the ends of the lines refer to the hank of the slubbing used. (From Foster [23].)*

Therefore, it appears that with the older processes with a large number of roving stages, the equipment did not have to be kept in as good condition as with the new, high-draft systems.

Some of these defects cause what is termed a "latent wave," which is a wave so short that the overlapping of fibers covers up the wave. This type of wave is actually a variation of fiber ends, and is shown up as a mass variation in the next process. It was discussed by Gregory [31] and Foster [25, 26].

A review of the work on this subject published in 1950 and 1951 was given by Hargreaves [35].

Theories of Fiber Movement in Drafting

The theory of fiber movement in cotton drafting was first approached by Balls [5], who analyzed the situation qualitatively and blamed the drafting wave on the floating fibers, those held by neither set of rolls. Foster [22] examined the conditions of drafting, and concluded that the motion of floating fibers could be expected to produce a drafting wave. Measurements of the amplitude and frequency of the wave showed that both are very variable, so that defects in positively driven parts could not cause it. However, roller slippage was a possibility, but this was discounted by experiments which showed that the amplitude increases with increases in roller setting. Therefore, it was concluded that the floating fibers do cause the drafting wave.

After this work, Foster [24] spent several years evolving a theory of fiber motion which he called a first step in explaining how fibers move in the draft zone. The basis for the theory is the deviation from perfect drafting caused by the floating fibers. In the conception of perfect drafting, fibers are pulled forward as they reach the front-roll nip. When floating fibers are present, they are pulled forward before their normal time by the longer fibers, thus producing a thick spot followed by a thin spot.

The basic concept in his theory is the "moving boundary hypothesis." He considered that at any

instant there is a boundary in the draft zone at which fibers change speed. Because of the action of the floating fibers, this boundary has a reciprocating motion, within limits, in the draft zone.

The motion of this boundary may be stable or unstable. Calculations made on the stable type of system do not agree with the measurements made on cotton strands, whereas those on the unstable system do. In the unstable system, whenever the forward velocity of the boundary exceeds the speed of the back rolls, the strand breaks. These breaks may be complete, or, if they are bridged by long fibers, they may be partial. This motion may not be in phase across the width of the strand, so the resultant strand has a composite wave. Indications are that the drafting of cotton follows this type of action.

He studied four different concepts of the type of movement. The one that works out best assumes that the distance of the moving boundary from the front limit is proportional (K is the factor) to the total length of fiber held by the front rolls and projecting into the draft zone. When drafting starts, the moving boundary starts backwards, reaches its back limit, and starts to return. When it exceeds the speed of the back rolls, a break occurs, and drafting starts again when new fibers reach the front limit. Under this hypothesis, the calculated wave lengths agree well with experimental values for two cottons, but disagree for a third. An extension of the same hypothesis takes care of the third. The amplitudes (added variance) follow the laws of change with draft that were found empirically.

The good agreement between the theory and experiment obtained with this hypothesis strongly suggests that the picture of the drafting wave as a succession of breaks is correct. However, the theory is not yet complete. The factor K has to be found empirically. Also, these hypotheses are assumptions about the motion of fibers, and a more fundamental explanation of movement in terms of forces on the fibers is in order.

There have been other theories of drafting. Some earlier ones like Hertel's [38], which was discussed by Löfgren [43], have only touched the problem. The others are in the field of woolen and worsted drafting. See, *e.g.*, Ginsberg *et al.* [29], Cox [14], Cox and Ingham [15], Green and Ingham [30], and Hannah [33]. Foster [24] studied most of these, and discussed ([24], p. T366) each of their approaches; he concluded that none of their theories applies to the drafting of cotton.

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Book Reviews

Chemistry of Carbon Compounds. Volume I, Part B; Aliphatic Compounds. Edited by E. H. Rodd. Texas, Elsevier Press Inc., 1952. xvi + 684 (779–1462) pp., 23 figures. Price, $17.50.

(*Reviewed by John G. Hildebrand*)

This book comprises the second part of the first volume, in a comprehensive five-volume treatise on the *Chemistry of Carbon Compounds,* and completes the section on Aliphatic Compounds. It is divided into twelve chapters which begin with Hydroxy- and Amino-monocarboxylic Acids and related compounds and follows the Richter Classification through Aldehydo- and Keto-acids, Carbonic Acid and Dicarboxylic Acids to Tri-, Tetra-, Penta-, Hexa- and Polyhydric Alcohols and their oxidation products. Sections devoted to Complex Carbohydrates, Proteins and Enzymes complete the present volume. Rubber and rubber-like high molecular weight substances, because of their relationship to the Terpenes, have been deferred to the second volume of the series which deals with the Alicyclic Compounds.

The presentation is factual, with emphasis on preparative methods, occurrence, reactions, and physical properties of the parent substance and numerous derivatives. One or more of thirteen distinguished contributors have prepared the individual chapters or essays.

The thorough documentation established in Part A of Volume I is carried out in Part B. Approximately one third of the references cited have been selected from the literature of the last 10 to 15 years. References to review articles, patent literature, specialized works such as "Organic Syntheses" and to other volumes in this series frequently occur. An Index of 70 pages containing approximately 7,000 entries is included. A sheet of "Addenda and Corrigenda" to Volume I, Part A is supplied with Part B.

While certain minor objections might be cited in connection with this book, your reviewer feels that they are likely to take the form of personal preferences; a kind of second guessing or "what might have been," now that a diligent and competent group of workers have presented the result of their labor. In view of the magnitude of the undertaking and the excellence of the result thus far, in the presentation of a general reference work in the field of organic chemistry, we believe it is unnecessary to attempt to determine just where this book fails to meet a particular idea of perfection. In our opinion, Part IB is a worthy companion to Part IA and may be recommended to practicing organic chemists as a valuable addition to their reference library.

Encyclopedia of Surface Active Agents. J. P. Sisley (P. J. Wood, translator). New York, Chemical Publishing Company, Inc., 1952. 540 pages. Price, $15.00.

(*Reviewed by Hillary Robinette, Jr.*)

The author and translator of *Encyclopedia of Surface Active Agents* have done a real service to those interested in surface active agents, particularly textile chemists who use them most.

Part one deals with the general aspects of surface active agents, their properties, applications and methods of manufacture. It outlines a very efficient system of classification of all surface active agents into many chemical subheadings under the three main captions of anion-active, cation-active, and non-ionic compounds. This method greatly facilitates an understanding and simplification of the description of individual products covered in part two of the book. The many applications of surface active agents in the textile industry, leather industry, as industrial and household detergents, in the treatments of metals, in surface coatings, in paper manufacture as flotation agents, for breaking emulsions in fats splitting, in soap making, in the rubber industry, in fruit and vegetable processing, in cosmetics, in inks, in cutting and soluble oils, are adequately described. A listing of the commercial products under each coded type of surface active chemical is contained.

It is the opinion of the reviewer that the greatest contribution made by Professor Sisley and Dr. Wood in this book is the attempt to systematically classify surface active agents by an extremely flexible symbol coding device. It is believed that the text is not suitable for the beginner in the field, since it only superficially treats with the fundamental properties of surface active agents. It will be found extremely

useful by the industrial specialist and by others as a source book of surface active agents manufactured in America and abroad.

Styrene, Its Polymers, Copolymers and Derivatives. R. H. Boundy and R. F. Boyer. New York, Reinhold Publishing Corporation, 1952. xxii + 1304 pages. Price, $20.00.

(*Reviewed by Joseph Fisher*)

The publication of this book is a result of the phenomenal growth in both research and production of styrene and its derivatives in the past two decades. It is a remarkable achievement that a chemical, commercially insignificant twenty years ago, could be produced at the rate of several hundred million pounds a year so soon after production was attempted.

The book, No. 115 in the A. C. S. Monograph Series, contains twenty-two chapters by various authors covering, in detail, the history, chemistry, and technology of monomeric styrene, polystyrene, and the many and increasingly important styrene derivatives. The editors have made available in one volume the large mass of reference material which will be extremely useful to the technologist or the research chemist seriously interested in the subject.

As might be expected in a multi-authored work, the level of the presentation varies in different chapters. Thus, Simha and Wall's excellent discussion of copolymerization (Ch. 20) follows a less critical chapter surveying German practice, the salient points of which might more appropriately have been reported in other chapters of the book dealing with the production and processing of styrene and polystyrene; this has, in fact, also been done to some extent. The book is purposely repetitive in many places so that the reader with limited interests will not miss basic information. Another valuable feature is the thorough presentation of the complex and controversial facts concerning polymerization kinetics, structure, and molecular weight of polystyrene.

It is unfortunate that all of the chapters in the book are not equally current. The editors have recognized this problem and an Appendix has been added which presents, for some chapters, more recent work of the past several years. The Addenda might have been more conveniently placed at the end of the proper chapters but the references are available for those interested. The survey of patent literature on styrene copolymers, complete to 1951, should be a most valuable aid to research workers entering the field.

It is to be hoped that the high cost of this volume will not be a deterrent to its addition to the library of the individual chemist.

Fundamental Principles of Polymerization—Rubbers, Plastics, and Fibers. G. F. D'Alelio. New York, John Wiley & Sons, Inc., 1952. 517 pages. Price, $10.00.

(*Reviewed by Hillary Robinette, Jr.*)

For those interested in the present status of the developments in the important field of polymer chemistry and polymer physics, Dr. D'Alelio has indeed contributed a volume that will be found extremely useful. As the title indicates, the book treats the fundamental principles of polymerization. The minimum fundamental knowledge necessary for a scientist working in the field of polymers is included in this volume.

The reader becomes thoroughly familiarized with the manner in which high polymers are made and the reasons why they have the properties which make them useful in the wide range of industrial applications to which they are put.

Emphasis is placed on the description of molecular processes which take place during polymerization, and of the molecular properties which make high polymers behave as they do, as rubbers, plastics, and fibers. Physical characteristics, such as viscosity, swelling, elasticity, tenacity and strength, are explained.

The book is well written, well documented, and well illustrated. *Fundamental Principles of Polymerization* may well be used as a text book in the class room and by industrial chemists and physicists as well.

The author and subject indexes are adequate.

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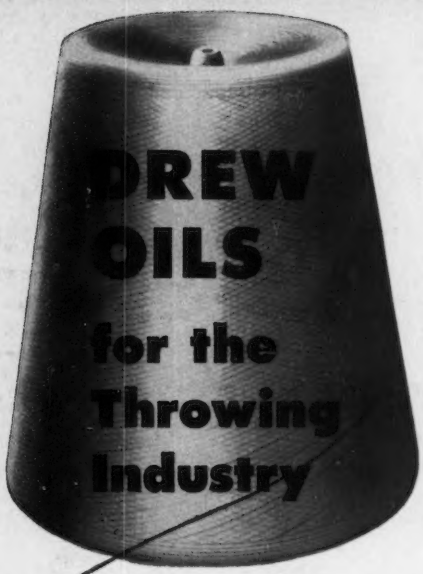

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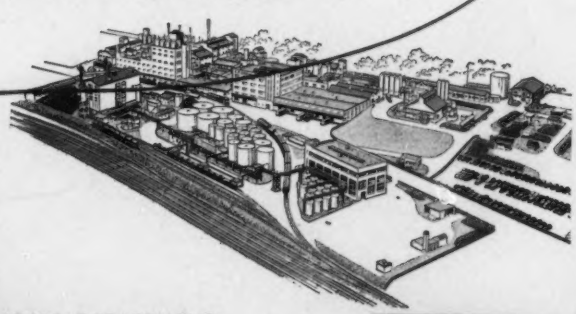

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